#### Remedial Action Contract for Remedial Response, Enforcement Oversight, and Non-Time Critical Removal Activities at Sites of Release or Threatened Release of Hazardous Substances in EPA Region VIII

U.S. EPA Contract No. EP-W-05-049

# Sampling and Analysis Plan, General Property Investigation Libby Asbestos Site, Operable Unit 4

Work Assignment No.: 229-RICO-08BC

Libby Asbestos Superfund Project,

**OU4 Remedial Investigation/Feasibility Study** 

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## **April 23, 2010**

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# **Acronyms**

A&E architecture and engineering contractor
AHERA Asbestos Hazard Emergency Response Act
ASTM American Society for Testing and Materials

BNSF Burlington Northern Santa Fe CAR corrective action request

CDM Federal Programs Corporation

COC chain-of-custody

CFR Code of Federal Regulations

CSHASP comprehensive site health and safety plan

CSS Contaminant Screening Study

CUA common-use area
DI detailed investigation
DQOs data quality objectives
EDD electronic data deliverable

EPIF exterior property inspection form
EPA U.S. Environmental Protection Agency
ERS Environmental Resource Specialist

FPM field planning meeting

eFSDS electronic field sample data sheet

FTL field team leader

GPI General Property Investigation
GPS global positioning system
HASP health and safety plan

HVAC heating, ventilation, and air conditioning

ID identifier

IDW investigation-derived waste IFF information field form

IPIF interior property inspection form

ISA interior surface area

LA Libby Amphibole asbestos LC laboratory coordinator

LUA limited-use area

NIOSH National Institute for Occupational Safety and Health

NUA non-use area

OIF occupant information form

OSHA Occupational Safety and Health Administration

OU operable unit

PLM polarized light microscopy

PLM-VE polarized light microscopy – visual estimation

PPE personal protective equipment

QA quality assurance QC quality control

RAWP Response Action Work Plan RI remedial investigation SI screening investigation

Site OU4 of Libby Asbestos Superfund Site



SAP sampling and analysis plan SOP standard operating procedure SRC Syracuse Research Corporation

SUA specific-use area

TEM transmission electron microscopy

TL task leader

VVEF visual vermiculite estimation form

W.R. Grace W.R. Grace Company

Zonolite Universal Zonolite Insulation Company



# Section 1 Introduction

This document serves as the General Property Investigation (GPI) sampling and analysis plan (SAP) for operable unit (OU) 4 (Site) of the Libby Asbestos Superfund Site. This SAP outlines activities that will be implemented to screen properties for Libby Amphibole asbestos (LA) or LA source materials and to determine the extent of any required removal action at each property selected for investigation. These activities include verbal interviews, interior and exterior visual inspections, and soil and bulk material sampling.

This SAP contains all the elements required for both a field sampling plan and quality assurance (QA) project plan. This SAP was developed in accordance with the Environmental Protection Agency Requirements for Quality Assurance Project Plans, EPA QA/R-5 (EPA 2001), and the Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G4 (EPA 2006).

The purpose of this SAP is to describe the sampling objectives, locations, measurement methods, and data quality objectives (DQOs) to support residential, commercial, and industrial response actions. The SAP is organized as follows:

Section 1 - Introduction

Section 2 - Site Background

Section 3 - Data Quality Objectives

Section 4 - Sampling Program

Section 5 - Laboratory Operations

Section 6 - Assessment and Oversight

Section 7 - Data Review and Verification

Section 8 - References

The process detailed within this SAP will be independent of the EPA contractor completing the activities, herein referred to as the architectural and engineering contractor (A&E).

#### 1.1 Objectives

This section defines the objectives of the GPI program and the intended use of data.

There are two main objectives to the sampling program described in this SAP:

- 1. Collect data to confirm the presence/absence of LA and/or LA source materials at residential, commercial, industrial, and public properties within OU4.
- 2. Collect data to determine the extent of removal activities required at properties within OU4 where previous investigations indicate the presence of LA and/or LA source materials.



# 1.2 Project Schedule and Deliverables

Sampling activities are planned to begin in April 2010. It is estimated that over 700 properties in OU4 have had no previous investigation completed. In addition, more than 400 properties in OU4 have been identified as requiring further investigation and removal activities. The overall schedule for implementing the sampling program described in this SAP will be determined by the annual clean-up goals.



# Section 2 Site Background 2.1 Site Location

The Site is located within Sections 3 and 10, T30N, R31W of the Libby Quadrangle in Lincoln County, Montana (Figure 2-1). The Site includes homes and other businesses that may have become contaminated with LA as a result of the vermiculite mining and processing conducted in and around the City of Libby (Figure 2-2).

The Site has been subdivided into eight OUs (Figure 2-3) to facilitate a phased approach to cleanup:

- OU1. The former export plant is defined geographically by the property boundary
  of the parcel of land that included the former export plant and nearby impacted
  areas.
- OU2. OU2 includes areas impacted by contamination released from the former screening plant. These areas include the former screening plant, the adjacent Flyway property, the Highway 37 right-of-way adjacent to the former screening plant and Rainy Creek Road, and privately-owned property. The Kootenai Bluff Subdivision area (former railroad loading station area), located directly across the Kootenai River from the former screening plant, has been removed from OU2 and is now part of OU4.
- OU3. The mine OU includes the former vermiculite mine and the geographic area (including ponds) surrounding the former vermiculite mine that has been impacted by releases from the mine, including Rainy Creek and the Kootenai River. Rainy Creek Road is also included in OU3. The geographic area of OU3 is primarily based upon the extent of contamination associated with release from the former vermiculite mine.
- OU4. OU4 is defined as residential, commercial, industrial (not associated with former W.R. Grace Company [W.R. Grace] operations), and public properties, including schools and parks in and around the City of Libby, or those that have received material from the mine not associated with W.R. Grace operations.
- OU5. OU5 is defined geographically by the parcel of land that included the former Stimson Lumber Company. OU5 is bounded by the high bank of Libby Creek to the east, the Kootenai River to the north, and residential/commercial/industrial property within OU4 to the south and west. This OU is approximately 400 acres in size and is currently occupied by various vacant structures/buildings as well as multiple operating businesses (lumber processing, log storage, excavation contractor, etc.). Within the OU5 boundary is the Libby Groundwater Superfund Site, which is not associated with the Libby Asbestos Superfund Site.



- OU6. Owned and operated by the Burlington Northern and Santa Fe Railroad (BNSF), OU6 is defined geographically by the BNSF property boundaries from the eastern boundary of OU4 to the western boundary of OU7 and extent of contamination associated with the rail yard.
- OU7. Approximately 20 miles west of downtown Libby, the Troy OU includes all residential, commercial, and public properties in and around the town of Troy, Montana.
- OU8. OU8 is comprised of the United States and Montana State Highway right-ofways within the OU4 and OU7 boundaries.

#### 2.2 Site History

Vermiculite was discovered 7 miles northeast of Libby, Montana in 1881 by gold miners. In the early 1920s, Edward Alley began initial mining operations on the vermiculite ore body located approximately 7 miles northeast of Libby. Full-scale operations began later that decade under the name of the Universal Zonolite Insulation Company (Zonolite). This ore body contains a solid solution series of amphibole asbestos fibers with compositions including tremolite, richterite, and winchite (herein referred to as LA) as defined by Meeker *et al.* (2003). Unlike chrysotile asbestos, LA has never been used commercially on a wide scale. During the mine's operating life, while vermiculite was used in a variety of products (including insulation and construction materials, as a carrier for fertilizer and other agricultural chemicals, and as a soil conditioner), LA was considered a byproduct of little or no value.

The vermiculite ore was mined using standard strip mining techniques and conventional mining equipment. The ore was then processed in an onsite dry mill to remove waste rock and overburden material. Once processed, the ore was transported from the mine to the former screening plant, where the ore was sorted into five size ranges. After the sorting process, the material was shipped to various locations across the United States, for either direct inclusion in products or for "expansion" prior to use in products. Expansion (also known as "exfoliation" or "popping") was accomplished by heating the ore, usually in a dry kiln, to approximately 2,000 degrees Fahrenheit. This process explosively vaporizes the water contained within the phyllosilicate structure causing the vermiculite to expand by a factor of 10 to 15. This produces the vermiculite material most commonly sold as a soil amendment for gardens and greenhouses.

In Libby, operations handling this material occurred at four main locations: the mine and mill located on Rainy Creek Road on top of Zonolite Mountain; the former screening plant and railroad loading station located at the intersection of Highway 37 and Rainy Creek Road and directly across the Kootenai River, respectively; the former expansion/export plant (the former export plant) located immediately west of Highway 37 where it crosses the Kootenai River; and at the former expansion plant located at the end of Lincoln Road, near 5th Street. The Lincoln Road Expansion Plant



went offline sometime in the early 1950s. In 1963, W.R. Grace purchased Zonolite and continued vermiculite mining operations in a similar fashion. In 1975, a wet milling process was added that operated in tandem with the dry mill until the dry mill was taken offline in 1985. The wet milling process was added to reduce dust generation of the milling process. Expansion operations at the former export plant ceased in Libby sometime prior to 1981, although this area was still used to bag and export milled ore until mining operations concluded in 1990. Before the mine closed in 1990, Libby produced about 80 percent of the world's supply of vermiculite.

Since 1999, Environmental Protection Agency (EPA) Region 8 has been conducting sampling and cleanup activities to address highly contaminated areas in the Libby valley. The EPA inspection was initiated in response to media articles, which detailed extensive asbestos-related health problems in the Libby population. While at first the situation was thought limited to those with direct or indirect occupational exposures, it soon became clear that there were multiple exposure pathways and many persons with no link to mining-related activities were affected.

#### 2.3 Occurrence of LA

Typically, the LA contamination found in the Libby valley comes from one or some combination of "primary" sources: vermiculite mining wastes, vermiculite ores, vermiculite processing wastes, bulk residuals from vermiculite processing, LA-containing rocks, or vermiculite insulation. Asbestos from these primary sources has been found in interior building dust samples and local soils, which in turn act as secondary sources. To date, EPA's goal has been to find and identify areas with elevated levels of LA (the primary sources) and to remove them. EPA has conducted removal of contaminated soil at the former export plant location, the former screening plant and adjacent properties, and residential, commercial, and public properties with LA source materials present. Removal actions have also been performed at three schools in Libby.

Cleanup work in Libby is ongoing and includes the removal of LA-containing media that include: vermiculite-containing materials (including vermiculite insulation and building materials with vermiculite additives), soil, and dust from residential, commercial, and industrial properties. The vermiculite encountered within structures/buildings is typically found in attics and exterior walls where it is used for insulation. In some cases, vermiculite insulation is found in interior and exterior walls due to sifting from the attic. In rare cases, vermiculite is found as an additive in building materials such as plaster, mortar, and concrete. The LA-contaminated soil encountered is generally due to vermiculite used as a soil amendment in flowerbeds and gardens, for leveling of low spots, and for backfilling of utilities. LA-contaminated dust occurs inside structures due to vermiculite insulation leaking into the living spaces from the attic or walls, and/or LA being tracked inside from the outdoor source locations discussed above.



#### 2.4 Summary of Previous Investigations

Historical inspections at the Site include the Phase 1 and Phase 2 investigations, contaminant screening study (CSS), and the ongoing remedial investigation (RI).

The Phase 1 sampling program, initiated in early 2000, was designed as a rapid pilot-scale inspection to:

- Determine whether or not airborne asbestos levels in Libby required time-critical action to protect public health
- Obtain data on asbestos levels in potential source materials
- Identify the most appropriate analytical methods to screen and quantify asbestos in source materials

The Phase 2 sampling program began in March 2001 and was designed to refine human exposure and health risk estimates through collection of systematic data on asbestos levels in air and other media and identification of sources of airborne asbestos. A summary of the findings of the Phase 1 and 2 studies is presented below:

- Asbestos occurs in ore and processed vermiculite obtained from the mine site located outside the City of Libby.
- Asbestos fibers of the type that occur in vermiculite ore from the mine site are hazardous to humans when inhaled.
- Asbestos fibers that are characteristic of those that occur in materials from the Libby mine are present in a variety of different source materials at residential and commercial locations in and around Libby.
- Disturbance of asbestos-contaminated source materials can result in exposure to respirable asbestos fibers in air.
- The concentrations of fibers in air generated by disturbance of source materials may exceed Occupational Safety and Health Administration (OSHA) standards for acceptable occupational exposures, and estimated excess cancer risks can exceed EPA's typical risk range by an order of magnitude or more.

The CSS inspection, which was part of OU4 RI activities, was initiated in early 2002 and employed a combination of visual inspections, verbal property owner interviews, and outdoor soil sampling to screen each property in the study area for the presence of potential sources of LA in areas where exposure is most likely to occur.



The primary objective of the CSS was to determine the presence or absence of potential LA sources at each property within the study area. Additional objectives included:

- Identification of properties that require remediation (i.e., contain primary sources)
- Identification of properties that require further inspection (i.e., contain or have indicators of secondary sources)
- Quantification of relative LA abundance in soils
- Identification of characteristics of properties that may increase chances of exposure to LA
- Identification of characteristics of properties that may aid in development of remedial decisions
- Determination of spatial trends

Results of the Phase 1 and CSS inspections are currently used to determine which properties would require additional investigation and subsequent cleanup activities. The status for each property, as of 2006, was summarized in the 2006 CSS Final Technical Memorandum (CDM 2006a). It is estimated that over 700 properties within OU4 were not inspected during the Phase 1 and CSS sampling programs.



# Section 3

# **Data Quality Objectives**

The DQO process, based on scientific methods, is a series of planning steps that are designed to ensure that the type, quantity, and quality of environmental data used in decision-making are appropriate for the intended purpose. The DQOs presented in this section were developed in accordance with EPA guidance (EPA 2006).

The DQO process specifies project decisions, the data quality required to support those decisions, specific data types needed, data collection requirements, and analytical techniques necessary to generate the specified data quality. The process also ensures that the resources required to generate the data are justified. The DQO process consists of seven steps; output from each step influences the choices that will be made later in the process. These steps include:

- 1. State the problem
- 2. Identify the decision
- Identify the inputs to the decision
- 4. Define the study boundaries
- 5. Develop a decision rule
- 6. Specify tolerable limits on decision errors
- 7. Optimize the design

#### 3.1 Step 1 – State the Problem

The purpose of this step is to describe the problem to be studied so that the focus of the investigation will be unambiguous.

Several property investigation field efforts have been completed for the Site, including the Phase 1 and Phase 2 investigations and the CSS. A number of properties were not inspected during these investigations due to various circumstances including refusal of access, inability to contact property owners, and incomplete county parcel information. Consequently, there are a number of properties where it is unknown whether LA contamination exists. In addition, some historical investigations were performed using techniques that differ from current site investigation protocols (e.g., 5-point composite sampling, high-traffic area visual inspections, semi-quantitative estimation of vermiculite, etc.).



This GPI SAP was developed to reflect current investigation and sampling methods. The GPI process is divided into two distinct phases; screening investigation (SI) and detailed investigation (DI). SIs are intended to screen properties that have not undergone previous investigation, while DIs are performed at properties where a removal trigger has been identified. The SI and DI phases will be combined into a single property visit when possible. Therefore, the overall GPI sampling program described in this SAP is designed to:

- Determine if LA and/or LA source materials are present at residential, commercial, industrial, or public properties within OU4.
- Determine the extent of LA contamination on each property if LA or LA source materials are present.

This SAP describes the sampling and inspection procedures that will be used to collect data of sufficient quality and representativeness to evaluate each of these items.

## 3.2 Step 2 - Identify the Decision

This step identifies what questions the investigation will attempt to resolve and what actions may result. The principal study questions and possible alternative actions are as follows:

Response Item Evaluated	Principal Study Question	Alternative Actions
Screening Investigation		
	Is vermiculite-containing material present in buildings?	<ul><li>Terminate SI phase and transition to DI phase</li><li>Take no action</li></ul>
Data with a sale	Is vermiculite visible in surface soils?	<ul> <li>Terminate SI phase and transition to DI phase if vermiculite is found in SUAs</li> </ul>
Determine if LA or LA source materials are present on individual properties.		<ul> <li>For CUAs and LUAs, document location of surface soils that contain vermiculite and isolate these areas for discrete sampling</li> <li>Take no action</li> </ul>
	Is LA detected at levels ≥1% in any surface soil samples collected from individual properties?	<ul> <li>Document location of LA- contaminated surface soils and transition to DI phase</li> <li>Take no action</li> </ul>
Detailed Investigation		
Determine the extent of LA contamination on individual	Is vermiculite insulation present in property buildings?	<ul> <li>Sketch and document location and extent of vermiculite for removal action</li> <li>Take no action</li> </ul>
properties if LA or LA source materials are present.	Is LA detected in friable building materials (e.g., plaster) that contain vermiculite additives?	<ul> <li>Collect samples, sketch and document location and extent of LA-contaminated building materials for removal action</li> <li>Take no action</li> </ul>



Response Item Evaluated	Principal Study Question	Alternative Actions
	Is LA detected at concentrations ≥5,000 s/cm² in indoor dust from any one previously collected dust sample from individual properties?	<ul> <li>Sketch and document location of living space with LA-contaminated indoor dust for removal action</li> <li>Take no action</li> </ul>
	Is vermiculite visible in surface soils?	<ul> <li>Sketch and document location and extent of vermiculite-containing soil for removal action</li> <li>Take no action</li> </ul>
	What is the extent of LA contamination in surface soils?	<ul> <li>Collect samples, sketch and document location and extent of LA-contaminated soil for removal action</li> <li>Take no action</li> </ul>

CUA – common-use area
DI – detailed investigation
LA – Libby Amphibole asbestos
LUA – limited-use area
s/cm² – structures per square centimeter
SI – screening investigation
SUA – specific-use area
≥ – greater than or equal to

## 3.3 Step 3 - Identify the Inputs to the Decision

The purpose of this step is to identify the information and measurements that need to be obtained to resolve the decision statements. The information needed to resolve the principal study questions are summarized in Table 3-1.

## 3.4 Step 4 - Define the Boundaries of the Study

This step specifies the spatial and temporal boundaries of this investigation.

#### 3.4.1 Spatial Bounds

The information gathered to answer the objectives will be collected from properties within the boundaries of OU4 and outside existing OUs as directed by EPA (Figure 2-3). The vertical spatial boundaries extend from the highest point at a property, approximately two stories, to the depth of soil samples collected, approximately 6 inches below ground surface.

#### 3.4.2 Temporal Bounds

For each property, the temporal boundaries of this investigation include the time from when an SI begins to the time it is determined LA or LA source materials do not exist on the property or when a DI is complete.

#### 3.5 Step 5 - Develop Decision Rules

The purpose of this step is to describe the method that EPA will use to determine if the data collected indicate acceptance and the resulting decision applied when acceptance is not obtained. The principal study question, inputs to resolve study questions, action levels, and decision rules are summarized in Table 3-2.



**Table 3-1 Summary of Inputs to Resolve Study Questions and Use of Information Acquired from Inputs** 

Principal Study Question	Input to Resolve Question	Use of Input to Resolve Question
Screening Investigation	440011011	
Is vermiculite-containing material present in buildings?	Visual Inspection	For each property undergoing an SI, attics, living spaces, walls, and understructures will be inspected for vermiculite-containing material to the extent possible. The results of the inspection will be used to determine if an LA source material is present within buildings at individual properties and result in removal action.
Is vermiculite visible in surface soils?	Visual Inspection	For each property undergoing a screening investigation, a semi-quantitative visual estimation inspection of vermiculite will be performed on surface soils throughout the entire property. The results of the visual inspection will be used to determine if LA source materials are present at individual properties and to determine if DI activities are required.
Is LA detected at levels greater than or equal to 1% in any surface soil samples collected from individual properties?	Soil Samples	For each property undergoing an SI, surface soil samples will be collected from use areas (e.g., SUAs, CUAs, LUAs, etc.). The results of the surface soil samples will be used to determine if LA contamination is present at individual properties and to determine if DI activities are required.
Detailed Investigation		
Is vermiculite insulation present in property buildings?	Visual Inspection	For each property undergoing a detailed investigation, a visual inspection will be performed within each building on the property. The results of the visual inspection will be used to determine the extent of LA source materials for removal planning.
Is LA detected in friable building materials (e.g., plaster) that contain vermiculite additives?	Bulk Material Samples	For each property undergoing a detailed investigation, bulk material samples will be collected from friable building materials that contain vermiculite. The results of the bulk material samples will be used to determine if LA contamination is present in the building materials at individual properties for removal planning.
Is LA detected at concentrations greater than or equal to 5,000 s/cm² in indoor dust from any one previously collected dust sample from individual properties?	Dust Samples	For each property where dust samples were collected during previous investigations, analytical results will be reviewed to determine if LA contamination is present in indoor dust at individual properties for removal planning. Dust samples will not be collected as part of this investigation.
Is vermiculite visible in surface soils?	Visual Inspection	For each property undergoing a DI, semi-quantitative visual estimation inspections for vermiculite will be performed on surface soils to determine the extent of LA source materials for removal planning.
What is the extent of LA contamination in surface soils?	Soil Samples	For each property undergoing a DI, additional surface soil samples may be collected from use areas (e.g., SUAs, CUAs, LUAs, etc.) to further determine the extent of LA contamination for removal planning.

CUA – common-use area
DI – detailed investigation
LA – Libby Amphibole asbestos

LUA – limited-use area SI – screening investigation SUA – specific-use area



**Table 3-2 Decision Rules** 

	Input to Resolve			
Principal Study Question	Question	Input Requirements	Action Level	Decision Rule
Screening Investigation				
Is vermiculite-containing material present in buildings?	Visual Inspection	Presence or absence of vermiculite via visual inspection	Presence of vermiculite	If open, non-contained, or migrating vermiculite is observed, the location will be documented and an interior DI will be performed.  If contained vermiculite is observed, the location will be
				documented.
				If vermiculite is not observed, take no action.
Is vermiculite visible in surface soils?	Visual Inspection	CDM-LIBBY-06	Detectable quantities of visible vermiculite as defined in CDM- LIBBY-06	If vermiculite is observed in surface soils within SUAs, the investigation activities will transition to the DI phase.
				If vermiculite is observed in surface soils only within non-SUAs, isolate these areas for discrete sampling.
				If vermiculite is not observed, take no action.
Is LA detected in individual surface soil samples collected from individual properties?	Soil Samples	Analysis: PLM-VE and PLM-Grav with project-specific modifications Reported Result: % LA AS: 0.2%	Greater than or equal to1% LA	If levels of LA greater than or equal to 1% are detected in surface soil samples, the investigation activities will transition to the DI phase.
				If levels of LA less than or 1% (≤1% LA, Trace, or non- detect) are detected in surface soil samples, take no action.
Detailed Investigation				
Is vermiculite insulation present in property buildings?	Visual Inspection	Presence or absence of vermiculite insulation via visual inspection	Presence of vermiculite	If vermiculite insulation is observed, the location will be documented for subsequent removal action.  If vermiculite insulation is not observed, take no action.
Is LA detected in friable building materials (e.g., plaster) that contain vermiculite additives?	Bulk Material Samples	Analysis: PLM by NIOSH 9002 Reported Result: % LA AS: Method defined as 1%, but qualitative estimates of LA present below 1% reported as less than 1% or ND	Any detectable LA	If LA is detected in any bulk material samples, the building material(s) that the bulk sample represents will be sketched/documented for subsequent removal action.  If LA is not detected in any bulk material sample, take no action.



**Table 3-2 Decision Rules (continued)** 

	Input to Resolve			
Principal Study Question	Question	Input Requirements	Action Level	Decision Rule
Detailed Investigation				
Is LA detected in indoor dust from previously collected individual dust samples from individual properties?	Dust Samples	Analysis: TEM by ASTM D5755 with project-specific modifications Reported Result: s/cm <sup>2</sup> AS: 1,000 per cm <sup>2</sup>	5,000 s/cm <sup>2</sup>	If LA is detected greater than or equal to 5,000 s/cm² in any dust sample, the living space that the dust sample represents will be sketched/documented for subsequent removal action.
				If LA is detected at levels less than 5,000 s/cm <sup>2</sup> in any dust sample, take no action.
Is vermiculite visible in surface soils?	Visual Inspection	CDM-LIBBY-06	Detectable quantities of visible vermiculite as defined in CDM- LIBBY-06	If vermiculite is observed in surface soils, the location will be sketched/documented for subsequent removal action.
				If vermiculite is not observed in surface soils, take no action.
What is the extent of LA contamination in surface soils?	Soil Samples	Analysis: PLM-VE and PLM-Grav with project-specific modifications Reported Result: % LA AS: 0.2% LA	Any detectable LA	If any detectable levels of LA are found in surface soil samples, the location will be sketched/documented for subsequent removal action.
				If LA is not detected in surface soil samples, take no action.

AS – analytical sensitivity
ASTM – American Society for Testing and Materials
DI – detailed investigation
LA – Libby Amphibole asbestos
NA – not applicable
ND – nondetect

PLM – polarized light microscopy s/cm² – structures per square centimeter SI – screening investigation SUA – specific-use area

TEM – transmission electron microscopy

% – percent



**Table 3-3 Limits on Decision Errors** 

Principal Study Question	Null Hypothesis	Type I Error Will Result in:	Type II Error Will Result in:
Screening Investigation			
Is open, non-contained, or migrating vermiculite containing material present in buildings?	Vermiculite is present in property buildings.	Determining that property buildings do not contain vermiculite when they actually do. This would result in no subsequent removal action and in turn, an increased risk to human health.	Determining that property buildings contain vermiculite when actually they do not. This would result in unnecessarily performing removal action planning and adds to investigation costs.
Is vermiculite visible in surface soils?	Vermiculite is present in surface soils.	Determining that surface soils do not contain vermiculite when they actually do. This may result in no subsequent exterior DI and in turn, an increased risk to human health.	Determining that surface soils contain vermiculite when they actually do not. This may result in unnecessarily performing an exterior DI and adds to investigation costs.
Is LA detected in surface soil samples collected from individual properties?	Surface soils are contaminated with LA.	Determining that surface soils are not contaminated with LA when they actually are. This may result in no subsequent exterior DI and in turn, an increased risk to human health.	Determining that surface soils are contaminated with LA when they actually are not. This would result in unnecessarily performing an exterior DI and adds to investigation costs.
Detailed Investigation			
Is vermiculite insulation present in property buildings?	Vermiculite insulation is present in property buildings.	Determining that property buildings do not contain vermiculite insulation when they actually do. This would result in no subsequent removal action and in turn, an increased risk to human health.	Determining that property buildings contain vermiculite insulation when they actually do not. This would result in unnecessarily performing removal action planning and adds to investigation costs.
Is LA detected in friable building materials (e.g., plaster) that contain vermiculite additives?	Friable building materials that contain vermiculite are contaminated with LA.	Determining that friable building materials that contain vermiculite are not contaminated with LA when they actually are. The LA-contaminated building material(s) would not be included in the removal action and in turn, an increased risk to human health.	Determining that friable building materials that contain vermiculite are contaminated with LA when they actually do not. This would result in unnecessarily including the building materials in the removal action and adds unnecessary costs to the investigation and removal.



Principal Study Question	Null Hypothesis	Type I Error Will Result in:	Type II Error Will Result in:
Is LA detected in indoor dust from previously collected dust samples?	Indoor dust is contaminated with LA.	Determining that indoor dust is not contaminated with LA when it actually is. The LA-contaminated living space would not be included in the removal action and in turn, an increased risk to human health.	Determining that indoor dust that contains is contaminated with LA when it actually is not. This would result in unnecessarily including an interior cleaning to the removal action and adds unnecessary costs to the investigation and removal.
Is vermiculite visible in surface soils?	Vermiculite is present in surface soils.	Determining that surface soils do not contain vermiculite when they actually do. The LA-contaminated soils would not be included in the removal action and in turn, an increased risk to human health.	Determining that surface soils contain vermiculite when they actually do not. This would result in unnecessarily including exterior excavation to the removal action and adds unnecessary costs to the investigation and removal.
What is the extent of LA contamination in surface soils?	Surface soils are contaminated with LA.	Determining that surface soils are not contaminated with LA when they actually are. The LA-contaminated soils would not be included in the removal action and in turn, an increased risk to human health.	Determining that surface soils contain are contaminated with LA when they actually are not. This would result in unnecessarily including exterior excavation to the removal action and adds unnecessary costs to the investigation and removal.

DI – detailed investigation LA – Libby Amphibole asbestos



#### 3.6 Step 6 - Specify Tolerable Limits on Decision Errors

The tolerable limits on decision errors, used to establish performance goals for the data collection design, are specified in this step.

Specific to performing SIs and DIs, two types of decision errors are possible:

- A Type I (false negative) decision error would occur if a risk manager decides that an inspection/sample does not contain vermiculite/LA above a level of concern, when in fact it is of concern.
- A Type II (false positive) decision error would occur if a risk manager decides that an inspection/sample does contain vermiculite/levels of LA above a level of concern, when in fact it does not.

EPA is most concerned about guarding against the occurrence of Type I errors, since an error of this type may leave humans exposed to unacceptable levels of LA.

EPA is also concerned with the probability of making Type II (false positive) decision errors. Although this type of decision error does not result in unacceptable human exposure, it may result in unnecessary expenditure of resources. Generally, EPA allows for a 20 percent false positive rate.

For the purposes of completing all seven steps of the DQO process, the null hypotheses and consequences of making an incorrect decision are summarized in Table 3-3. However, the gray region and tolerable limits on decision errors are not proposed because they are not applicable in this case.

Typically, Step 6 of the DQO process is useful to encourage careful design of decision rules by defining and integrating the errors that are acceptable based upon a myriad of integrated project management decisions such as reduction in risk to human health, implementability/practicability, and cost. As stated in the guidance document for development of DQOs: QA/G-4 (EPA 2006a), solely statistically generated tolerable limits on decisions errors are not necessary in certain cases provided that a line of reasoning (scientific justification) is presented that adequately defines acceptable limits or decision errors. This particular effort was put forth in the *Action Level/Clearance Criteria Technical Memorandum* (EPA 2003) for DQOs for the following sampling and inspection: (1) vermiculite in surface soils and property structures/buildings; (2) surface soil samples; and (3) indoor dust samples.

#### 3.7 Step 7 - Optimize the Design for Obtaining Data

This step identifies a resource-effective data collection design for generating data that are expected to satisfy the DQOs. The data collection design is described in detail in the remaining sections of this SAP and other site documents referenced in Section 4.



Referencing the *Action Level/Clearance Criteria Technical Memorandum* (EPA 2003) and data previously generated for the site, the DQOs have been designed to support the proposed SI and DI activities and represent the best possible project planning effort. However, in implementing the requirements contained in this SAP, unforeseen situations may arise or team members may find more efficient means to carry out some of the day-to-day activities. Therefore, team members are always afforded the opportunity to recommend optimization of the data gathering design.

Recommendations must come through proper channels (i.e., through the task leader [TL] or field team leader [FTL]) and documented using either a Libby Asbestos Project Record of Modification Form (provided in Appendix E) or an addendum to this SAP. All modifications or addendums must be approved prior to making the proposed changes.



# **Section 4 Sampling Program**

This section summarizes field activities that will be performed in support of general property investigations within OU4, and outside existing OUs as directed. This section also provides brief summaries of standard operating procedures (SOPs), including project-specific modifications where applicable and project-specific details not discussed in the SOPs. As previously mentioned, the GPI is designed to screen properties for the presence of LA or LA source materials and determine the extent of LA contamination for subsequent removal. The overall investigation process is outlined in Figure 4-1. The sampling program is divided into a two major phases:

- Screening Investigation (SI) (Section 4.2)
- Detailed Investigation (DI) (Section 4.3)

Specific details on each type of investigation are discussed below. For comprehensive health and safety information, field personnel will refer to the general and project-specific SOPs included in Appendix A. The *Comprehensive Site Health and Safety Plan* (CSHASP) (CDM 2006) and the A&E's site-specific health and safety plan (HASP) (CDM 2008) should be consulted to determine health and safety protocols for performing GPI work.

All sampling activities will be performed in accordance with this SAP. The SOPs and project-specific procedures to be employed are:

- Sample Custody (Modified CDM SOP 1-2)
- Packaging and Shipping of Environmental Samples (Modified CDM SOP 2-1)
- Guide to Handling of Investigation-derived Waste (IDW) (Modified CDM SOP 2-2)
- Field Logbook Content and Control (Modified CDM SOP 4-1)
- Field Equipment Decontamination at Non-Radioactive Sites (Modified CDM SOP 4-5)
- Control of Measurement and Test Equipment (CDM SOP 5-1)
- Project-specific SOP for Global Positioning System (GPS) Coordinate Collection and Handling (CDM-LIBBY-09)
- Project-specific SOP for Investigation Soil Sample Collection (CDM-LIBBY-05)
- Project-specific SOP for Visual Vermiculite Inspection (CDM-LIBBY-06)
- Project specific SOP for Electronic Field Data Capture and Production of Chain-of-Custody (COC) Records (CDM-LIBBY-15)



Table 4-1 indicates the current version of each SOP to be utilized as part of this SAP.

The following sections summarize field activities that will be performed during the implementation of the sampling investigation efforts described in this SAP.

Analytical methods for all samples collected in accordance with this SAP are discussed in Section 5.

**Table 4-1 Standard Operating Procedures** 

	Current Rev		
SOP	Revision	Title	Date
CDM SOP 1-2	5	Sample Custody	March 2007
CDM SOP 2-1	3	Packaging and Shipping of Environmental Samples	March 2007
CDM SOP 2-2	5	Guide to Handling Investigation-Derived Waste	March 2007
CDM SOP 4-1	6	Field Logbook Content and Control	March 2007
CDM SOP 4-5	7	Field Equipment Decontamination at Nonradioactive Sites	March 2007
CDM SOP 5-1	8	Control of Measurement and Test Equipment	March 2007
CDM-LIBBY-05	2	Soil Sample Collection at Residential and Commercial Properties	May 2007
CDM-LIBBY-06	1	Semi-Quantitative Visual Estimation of Vermiculite in Soils at Residential and Commercial Properties	May 2007
CDM-LIBBY-09	2	Global Positioning System (GPS) Coordinate Collection and File Transfer Process	July 2009
CDM-LIBBY-15	0	Completion of Electronic Surveys Using Mobile Surveyor	TBD

#### 4.1 Pre-Sampling Activities

Prior to beginning of field activities, a field planning meeting (FPM) will be conducted, any required training will be performed, and an inventory of equipment and supplies will be completed to determine procurement needs.

#### 4.1.1 Field Planning

Prior to beginning field activities, an FPM will be conducted by the A&E's investigation TL or FTL, which will be attended by the field team members conducting the work, a member of the A&E's QA staff, and a member of the A&E's health and safety staff. The agenda, prepared by the TL or FTL, will be reviewed and approved by QA and health and safety staff prior to the FPM. A field planning meeting agenda is provided in Appendix B. The FPM will briefly address and clarify:

- Documents governing fieldwork that must be on site
- Any changes in the governing documents
- Objectives and scope of the fieldwork
- Equipment and training needs



- Field operating procedures, schedule of events, and individual assignments
- Required quality control (QC) measures
- Health and safety requirements

During the FPM, copies of the agenda will be distributed and an attendance list will be circulated for signature. The agenda and the completed attendance list will be maintained in the A&E's project files. Additional meetings will be held when major changes to the documents governing fieldwork occur, or when the scope of the assignment changes significantly.

Field team members will perform the following activities before and during field activities, as applicable:

- Review and understand applicable governing documents
- Record appropriate levels of documentation regarding activities conducted
- Ensure coordination between key staff, such as the A&E's sample coordinator and the removal contractor
- Obtain required sample containers and other supplies
- Obtain, check, and calibrate field sampling equipment
- Obtain and maintain personal protective equipment (PPE)

#### 4.1.2 Field Team Training Requirements

Prior to starting work described in this document, any new field team member must complete the following, at a minimum:

- Read the CSHASP (CDM 2006) and site-specific HASP (CDM 2008)
- Attend an orientation session with the A&E's onsite health and safety officer
- Read and understand all relevant governing documents
- Attain OSHA 40-hour Hazardous Waste Operations and Emergency Response certification and relevant 8-hour refresher course certifications
- Attain respiratory protection course certification as required by 29 Code of Federal Regulations (CFR) 1910.134
- Attain asbestos awareness course certification as required by 29 CFR 1910.1001



- Complete training on sample collection techniques to the satisfaction of the GPI TL or FTL
- Complete training on identifying vermiculite and Libby mine-related materials to the satisfaction of the GPI TL or FTL

Documentation of trainings/certifications will be stored in the Libby project files located at the A&E's Denver office.

#### 4.1.3 Inventory and Procurement of Equipment and Supplies

An inventory of project-procured equipment and supplies will be conducted by the GPI TL or FTL prior to field work. Any additional required equipment or supplies will be procured. Acceptance of equipment, as pertinent, will be verified according to CDM SOP 5-1. The following equipment is required for sampling activities conducted under this SAP:

- Field logbooks
- Indelible ink pens
- Digital camera with memory card, as appropriate
- Sample paperwork and sample tags/labels
- Custody seals
- Plastic zip-top bags
- Soil sampling equipment
- GPS unit(s) (e.g., Trimble® Pathfinder Pro XRS or equivalent) and compass
- PPE as required by the CSHASP (CDM 2006) and site-specific HASP (CDM 2008)
- Cordless drill and scope
- Ladder
- Standard hand tools (screwdrivers, hammer, pry-bar, etc.)
- Toughbook laptop equipped with the Mobile Surveyor application (for completing forms/checklists)
- Measuring wheel/tape
- Land survey (hard copy)



## 4.2 Screening Investigation (SI)

This section describes the sampling methods and procedures that will be used for SIs. SIs are completed at properties that have not previously been characterized. The goal of completing an SI is to identify LA and/or potential sources of LA on a given property.

The major phases to an SI include:

- Property Selection and Initial Communication
- Verbal Interview
- Interior Inspection
- Exterior Inspection
- Screening Documentation
- All SI activities will follow a hot-stop process. The purpose of this process is to inspect a property until either the property has been fully characterized, or a removal trigger is identified. If a removal trigger is identified during an SI, the investigation will immediately transition to the DI phase.

Current removal triggers are:

- Visual confirmation of open, uncontained, or migrating vermiculite insulation.
- Visual confirmation of vermiculite in the indoor living space.
- Concentration of LA in an indoor dust sample greater than or equal to 5,000 LA structures per square centimeter (s/cm²) using American Society for Testing and Materials (ASTM) D5755, with project-modified Asbestos Hazard Emergency Response Act (AHERA) counting methods.
- Visual confirmation of vermiculite or other vermiculite mine-related materials in specific-use areas (SUA). An SUA is defined as a flowerbed, garden, former garden, planter, driveway, play area, or other defined area of a property likely to receive significant use and generally not covered with grass.
- Concentration of LA greater than or equal to 1 percent in SUAs or other yard soils by polarized light microscopy (PLM).



#### 4.2.1 Property Selection and Initial Communication

Prior to the start of investigation activities, a comprehensive list of properties requiring investigation will be compiled. Properties will then be selected for investigation based on geographic location, to maximize the efficiency of removal activities when required, and to ensure a systematic completion of investigation activities in each area of the Site.

Once a property has been selected for an SI, the property owner will be contacted to confirm willingness to participate. Information provided to the property owner at this time will include general details on the investigation and removal process, and a tentative time-frame for investigation and potential removal activities. A property will be placed in the queue for continued investigation activities once the owner has confirmed willingness to participate in the entire process. If the property owner is unwilling to participate with the complete process within the stipulated time-frame, the property will not be reconsidered for investigation until that geographic area is revisited. If the property owner refuses to participate in the investigation and removal process, a Property Refusal Form will be completed as discussed in the Response Action Work Plan (RAWP) (PRI 2010). Refusal information will be tracked in the project database in accordance with EPA data reporting requirements.

#### 4.2.2 Land Survey

A land survey will be conducted at each property identified for SI activities. Land surveys will include property boundaries to determine the limits of the property for which the removal is being conducted. Surveys will also include major physical and geographic features of the property (e.g., structures/buildings, trees, individual land use areas). The survey contractor will be a registered and licensed land surveyor in the State of Montana.

A survey will be ordered once a property owner has confirmed willingness to participate in the entire process. A hard copy of the survey will be used by the investigation team to mark soil sample locations and results, locations of visible vermiculite, and additional inspection information. Specific information to be captured by the investigation team is discussed in the following sections.

#### 4.2.3 Scheduling Screening Investigations

An SI will be scheduled for a time that is convenient for the property owner or tenant to be present and allow access to the interior of each structure/building at the property.

#### 4.2.4 Verbal Interview

Upon arriving at the property, the investigation team will meet with the property owner or tenants. The investigation team will provide the property owner/tenants with general details on the investigation and removal process, and tentative time-frame for future investigation steps, and, if required, removal activities. Additionally, the investigation team will note any pertinent anecdotal information provided by the



property owner for removal planning. This information may include scheduling requests, general property information, details on known contamination present at the property, etc. The investigation team will complete and obtain property owner signatures on a Consent for Access to Property form that will cover inspections to be performed immediately as well as any future investigation activities. Property owners will be notified prior to and may request to be present during any future investigation activities. Access information will be tracked in the project database in accordance with EPA data reporting requirements.

#### 4.2.5 Interior Screening Inspection

During SI activities, the purpose of the interior inspection is to fully inspect the interior of each structure/building on the property to confirm the presence or absence of LA source materials (e.g., vermiculite insulation, vermiculite-containing building materials). All inspection information is captured on the Interior Property Inspection Form (IPIF), and associated sketches. Occupant information and history for all occupied structure/building is captured on the Occupant Information Form (OIF). The interior of each structure/building on the property will be inspected during SI activities.

In addition, details collected during the interior inspection will be sufficient to support any future removal activities necessary and eliminate the need for subsequent investigation. All interior inspections will be completed to DI standards, in accordance with Section 4.3.5 of this SAP.

#### 4.2.6 Exterior Screening Inspection

Exterior screening inspections will be performed to identify potential LA source materials within the exterior soils of the property. All inspection information is captured on the Visual Vermiculite Estimation Form (VVEF), and associated sketches.

The purpose of the following sections is to detail procedures for conducting exterior inspections, which will be conducted by the A&E and will include the following activities:

- Visual Inspection
- Soil Sampling
- Exterior Inspection Documentation

#### 4.2.6.1 Visual Inspection

Visual inspection of exterior soils will be completed in accordance with CDM-LIBBY-06 with the following exception:

■ The number of point inspections to be completed per use area is defined in Table 4-2 (Screening Investigation).



**Table 4-2 Visual Inspection and Soil Sampling Protocol** 

Screening Investigation						
Area Type	Visual Inspection Protocol <sup>a</sup>	Soil Sampling Protocol <sup>b,c</sup> Maximum Area per Sample				
SUA <sup>d,e</sup> (flowerbed, garden, play area, etc.)	1 PI/100 ft <sup>2</sup>	use area type				
SUA <sup>d,e</sup> (driveway)	1 PI/200 ft <sup>2</sup>	use area				
CUA (yard, etc.)	1 PI/1,450 ft <sup>2</sup>	1 acre (43,560 ft <sup>2</sup> )				
LUA (field, pasture, etc.)	1 PI/7,260 ft <sup>2</sup>	5 acres (217,800 ft <sup>2</sup> )				
ISA (shed, carport, garage, crawlspace, etc.)	1 PI/100 ft <sup>2</sup>	use area				
NUA (wooded area, etc.)	No Inspection	No Sampling				
Detailed	Detailed Investigation					
Area Type	Visual Inspection Protocol <sup>a</sup>	Soil Sampling Protocol <sup>b,d</sup> Maximum Area per Sample				
SUA <sup>e</sup> (flowerbed, garden, play area, etc.)	1 PI/100 ft <sup>2</sup>	1,000 ft <sup>2</sup>				
SUA <sup>e</sup> (driveway)	1 PI/200 ft <sup>2</sup>	6,000 ft <sup>2</sup>				
CUA (yard, etc.)	1 PI/100 ft <sup>2</sup>	3,000 ft <sup>2</sup>				
LUA (field, pasture, etc.)	1 PI/500 ft <sup>2</sup>	15,000 ft <sup>2</sup>				
ISA (shed, carport, garage, crawlspace, etc.)	1 PI/100 ft <sup>2</sup>	use area				
NUA (wooded area, etc.)	No Inspection	No Sampling				

<sup>&</sup>lt;sup>a</sup>A minimum of 5 points will be inspected per use area regardless of size. <sup>b</sup>All soil samples are 30-point composites.

SUA – specific-use area

CUA - common-use area

LUA - limited-use area

NUA - non-use area

ISA – interior surface area

PI – point inspection ft<sup>2</sup> – square feet



<sup>&</sup>lt;sup>c</sup>Areas where vermiculite is observed within CUAs and LUAs will be segregated and sampled discretely.

dAreas where vermiculite is observed will not be sampled.

<sup>&</sup>lt;sup>e</sup>Multiple SUAs of the same type (e.g., flowerbeds may only be combined with other flowerbeds) within the same general area may be combined to form one sample area.

#### 4.2.6.2 Soil Samples

Soil samples will be collected during SIs in order to fully characterize each property. SI samples will only be collected if no removal trigger has been identified.

All SI soil samples will be collected in accordance with CDM-LIBBY-05 with the following exception:

- The maximum area that a single 30-point composite sample may include varies depending on the type of use area. Table 4-2 (Screening Investigation) defines the maximum area per soil sample.
- To ensure representativeness of the sampled use area, common use areas (CUAs) and limited use areas (LUAs) will be subdivided based on distinct physical barriers up to the maximum areas detailed in Table 4-2. For example, back yards will be sampled separately from front yards if they are separated by a fence or other site feature.
- Samples collected outside the computed property boundary (as defined on the land survey) will be associated with the property assuming its ownership if the difference between assumed and computed property boundary is less than 10 feet. If the difference is greater than 10 feet, samples will be associated with the actual property address where the samples were collected.

If vermiculite is observed in CUAs or LUAs during the visual inspection, and no removal action level is identified, those areas will be isolated from the remainder of the use area without visual vermiculite. Each CUA or LUA portion containing visible vermiculite will be sampled separately and in accordance with the sampling guidelines above.

#### 4.2.7 Screening Documentation

A VVEF will be completed for each SI conducted. In addition, an IPIF will be completed for each structure/building inspected as part of SI activities. An OIF will be completed for each occupied structure/building on the property. Example inspection forms are provided in Appendix C, and depict the data to be collected. All inspection forms will be completed electronically as described in CDM-LIBBY-15.

For exterior inspections, sample information and visual inspection results will be recorded on two separate property sketches. The land survey of the property will be utilized as the baseline for these sketches. Land surveys will include property boundaries, and will be provided prior to the start of investigation activities. Sample information and visual inspection results may be combined on one sketch if quality and clarity can be maintained.

In addition, investigation teams will review and provide feedback regarding GeoUnit-to-property relationships as discussed in the EPA Data Management Plan (EPA 2010).



#### 4.3 Detailed Investigation (DI)

This section describes the sampling methods and procedures that will be used to complete DIs. DIs are performed to capture additional information on a property to support cleanup activities. DIs are completed at properties that have undergone initial screening and display one or more removal triggers.

The following is a summary of field activities that will be performed by the A&E during the DI:

- Property Selection and Communication
- Land Survey
- Scheduling Investigations
- Review of Previously Collected Data
- Interior Inspection
- Exterior Inspection

#### 4.3.1 Property Selection and Communication

In general, property selection involves querying the project database for properties with specific contamination (i.e., indoor, outdoor, both), reviewing previously collected data, and clustering homes geographically to maximize the efficiency of the removal contractors. Within each geographic area, properties with children (i.e., occupants under 18 years of age) present will be prioritized to the extent possible.

If a property has undergone previous investigation activities (i.e., Phase I or CSS), or if soil sample results from the SI meet a current removal trigger, the property owner will be contacted to confirm willingness to participate. Information provided to the property owner at this time will include general details on the investigation and removal process, and a tentative time-frame for investigation and potential removal activities. A property will be placed in the queue for continued investigation activities once the owner has confirmed willingness to participate in the entire process. If the property owner is unwilling to participate with the complete investigation and removal process within the stipulated time-frame, the property will not be reconsidered for removal until that geographic area is revisited. If the property owner refuses to participate in the investigation and removal process, a Property Refusal Form will be completed as discussed in the RAWP (PRI 2010) and refusal information tracked in the project database.



### 4.3.2 Land Survey

A land survey will be conducted at each property identified for DI activities. Land surveys will include property boundaries to determine the limits of the property for which the removal is being conducted. Surveys will also include major physical and geographic features of the property (e.g., structures/buildings, trees, individual land use areas). The survey contractor will be a registered and licensed land surveyor in the State of Montana.

A survey will be ordered once a property owner has confirmed willingness to participate in the entire process. A hard copy of the survey will be used by the investigation team to mark soil sample locations and results, locations of visible vermiculite, and additional inspection information. Specific information to be captured by the investigation team is discussed in the following sections.

## 4.3.3 Scheduling Detailed Investigations

A DI will be scheduled for a time that is convenient for the property owner or tenant to be present and allow access to the interior of each structure/building on the property.

If a property has undergone SI and/or partial DI activities, and additional investigation activities are required, the property owner will be notified of the preferred time-frame to conduct these activities. If the property owner has requested to be present during all activities, a DI will be scheduled for a time that is convenient for them.

## 4.3.4 Previously Collected Data

Prior to arriving at a property, the investigation team will review all previously collected data in order to become familiar with property conditions. Previous data may include Phase 1, CSS, and/or SI data (including bulk material, soil, and dust sample results), and data collected during an Environmental Resource Specialist (ERS) initial response. A complete set of property-specific data will be obtained from the project database and maintained in the project file folder at the A&E's Libby, Montana office. All existing property documentation (Information Field Form [IFF], field sample data sheets [FSDSs], field logbook notes, ERS Initial Assessment Checklist, IPIF, etc.) will be reviewed to determine the general location of contaminated material.

In some cases, dust samples collected during previous investigations were not analyzed but were archived for potential future analysis. The investigation team will coordinate with the A&E sample coordinator to ensure that the necessary dust samples are retrieved from archive and analyzed.



### 4.3.5 Interior Detailed Inspection

Interior detailed inspections will be performed when previous investigation findings indicate either contamination is present or unknown within structures/buildings (e.g., house, garage, shed, barn) on the property. Also, interior detailed inspections will be completed during the SI phase if there have been no previous interior investigations completed at the property. A&E interior inspection activities include:

- Attic Inspection
- Living Space Assessment and Wall Inspection
- Understructure Inspection
- Bulk Material Samples, as required
- Interior Soil Samples, as required
- Interior Inspection Documentation

Interior inspections will be performed to determine the location and extent of contaminated materials within a structure/building. Information will also be collected regarding the general construction and condition of the structure/building, and access to contaminated materials. Interior inspections will include attic spaces, living spaces, and understructures (e.g., basement, cellar, crawlspace).

#### 4.3.5.1 Attic Inspection

Attic inspections will be completed when previous inspections indicate the presence of vermiculite insulation, or if the presence/absence of vermiculite could not be confirmed during previous investigations. Attic inspections will be limited to confirming the presence/absence of vermiculite insulation and collecting sufficient details to support removal activities. All attic spaces will be inspected until either vermiculite insulation is confirmed, or until the entire attic has been inspected and no vermiculite insulation is present.

Once vermiculite insulation is confirmed in an attic space, general details for the entire attic (including areas that share air space but do not contain vermiculite insulation) will be collected from that location and the inspection will cease. Detailed information about the attic space will be collected during the Interior Property Removal Evaluation as discussed in the RAWP (PRI 2010).

Attic details will be recorded on the IPIF and associated sketch(es) as discussed in Section 4.3.5.6.

#### 4.3.5.2 Living Space Assessment and Wall Inspection

Interior living spaces will be inspected to determine if vermiculite materials are present. Vermiculite may appear in living spaces as insulation that is leaking from the



attic or walls, or as an additive in building materials. Living space assessments will include inspecting all walls, all ceiling and wall penetrations (plumbing, heating, ventilation and air conditioning [HVAC] systems, electrical fixtures, cracks, gaps, etc.), and plaster/mortar materials. If vermiculite additives are identified within building materials, bulk material samples may be required as discussed in Section 4.3.5.4.

Based on previous investigation findings, small amounts of vermiculite insulation are likely to be present within wall cavities of structures/buildings that have vermiculite attic insulation. If vermiculite insulation is observed within the attic of a structure/building, it will be assumed that the walls below those attic sections contain some amount of vermiculite and those walls will not be inspected using intrusive methods. This will be noted within the interior inspection documentation as detailed in Section 4.3.5.6.

In addition, previous investigations have found vermiculite insulation used as primary insulation within wall cavities. All walls will be inspected to determine if vermiculite insulation is present, only if vermiculite was not observed within the attic or area above the walls. At least one location along each exterior and interior wall will be inspected. Non-destructive inspection methods will be utilized when possible. This will include removing electrical outlet and switch covers, and inspecting through other existing wall penetrations. Destructive inspection methods (i.e., drill/scope) will be used only when no existing penetrations are present. If destructive methods are required, care will be taken to minimize damage and inspections will be carried out within inconspicuous areas. The investigation team will seal all new penetrations with appropriate patching material.

Living space details will be recorded on the IPIF and associated sketch(es) as discussed in Section 4.3.5.6.

#### 4.3.5.3 Understructure Inspection

Building understructures will be inspected to determine if vermiculite materials are present. Vermiculite may appear in understructures as insulation that is leaking from the attic or walls, as additives in building materials, or as vermiculite in soil floors. Understructure inspections will include inspecting all ceiling and wall penetrations (plumbing, HVAC, electrical, cracks, gaps, fixtures, etc.), plaster/mortar materials, and inspecting soil floors. If the building understructure has a soil floor, a visual inspection will be completed per Section 4.3.6.1 of this SAP. If vermiculite is not observed within the soil floor, soil samples will be collected as discussed in Section 4.3.5.5.



Understructure details will be recorded on the IPIF and associated sketch(es) as discussed in Section 4.3.5.6. In addition to general details, the investigation team will make a determination as to the frequency the understructure is used. Understructures will be categorized as frequently used, infrequently used, or a combination of the two (for separate areas). Infrequently used understructures will include areas that are accessed on an irregular basis only, generally for maintenance purposes.

#### 4.3.5.4 Bulk Material Samples

Bulk material samples will be collected when vermiculite additives are identified within a building material, and only if that material is friable (i.e., able to be pulverized by hand).

Bulk material samples will be collected in compliance with AHERA sampling requirements provided in 40 CFR 763.86 (Appendix A).

#### 4.3.5.5 Interior Soil Samples

Soil samples will be collected from inside a structure only if significant soil areas are present (e.g., soil floor) where vermiculite was not observed during visual inspection. Individual flower pots/planters will not be sampled. Soil samples will be collected in accordance with Section 4.3.6.2.1 of this SAP.

#### 4.3.5.6 Interior Inspection Documentation

An IPIF (Appendix C) will be completed for each building/structure inspected as part of this SAP. IPIFs will be completed electronically as described in CDM-LIBBY-15.

Attic, living space, and understructure sketches will accompany each IPIF as appropriate. Sketches will include the details indicated in Table 4-3. Sketches will only be prepared for the levels/floors of the structure where LA source materials are observed and/or where samples are collected.

Investigation teams will collect digital photographs in accordance with Section 4.5.6 of this SAP. Photographs will include access points, interior hazards, pre-existing conditions, all areas where LA source materials are observed, and general interior photos.



#### **Table 4-3 Sketch Details**

#### **General Sketch Details**

To be included on all field sketches

- Property address
- BD number (for interior sketches)
- Inspection date
- Personnel (author)
- North arrow
- Scale (if applicable)
   Sketch description (e.g. attic, first floor, exterior analytical, exterior visual inspection, etc.)

<ul> <li>Sketch description (e.g. attic, first floor, exterior analytical, exterior visual inspection, etc.)</li> </ul>							
Interior Inspection Sketch Details							
Attic	Living Space		Understructure				
<ul> <li>Plan view/layout – including dimensions</li> <li>Types of insulation</li> <li>Depth of insulation</li> <li>Attic accesses - location and size</li> <li>Head space – structure crosssection</li> <li>Hazards (in attic and near access)</li> <li>Obstacles</li> <li>Joist – size and spacing</li> <li>Flooring (above and below joist)</li> </ul>	<ul> <li>Floor plan/layor</li> <li>Location of con materials</li> </ul>		<ul> <li>Soil samples – locations and results</li> <li>Visual inspection results</li> <li>Floor types – soil versus solid flooring</li> <li>Access – location and size</li> <li>Headspace</li> </ul>				
Exterior Inspection Sketch Details							
Analytical Sketch		Visual Inspection Sketch					
<ul> <li>Soil samples – locations and results         <ul> <li>Sample ID</li> <li>Location ID</li> </ul> </li> <li>Personal items within areas requiring removal</li> <li>Fence lines</li> <li>Underground utilities – if known</li> <li>Overhead utilities – if not shown on survey</li> <li>Location IDs for all structures on the property</li> <li>Type and condition of walkways and driveways</li> </ul>		<ul> <li>Location IDs, including Location IDs for all structures on the property</li> <li>Visual inspection results</li> <li>Personal items within areas requiring removal</li> <li>Fence lines</li> <li>Underground utilities – if known</li> <li>Overhead utilities – if not shown on survey</li> <li>Type and condition of walkways and driveways</li> </ul>					



### 4.3.6 Exterior Detailed Inspection

Exterior detailed inspections will be performed at properties where previously collected data indicates the presence of a current removal trigger. Exterior inspections are performed to further define the location and extent of contaminated material and to ensure that the entire property has been characterized. All inspection information is captured on the Exterior Property Inspection Form (EPIF), VVEF, and associated sketches.

A&E exterior inspection activities include:

- Visual Inspection
- Soil Sampling
- Exterior Inspection Documentation

#### 4.3.6.1 Visual Inspection

Visual inspection of exterior soils will be completed in accordance with CDM-LIBBY-06 with the following exceptions:

- The number of point inspections to be completed per use area is defined in Table 4-2 (Detailed Investigation).
- Areas that have been previously characterized and the presence of contamination has been confirmed (e.g., analytical results indicating the presence of LA) will not be visually inspected.
- In general, non-use areas (NUAs) are not inspected as part of this SAP. However, NUAs will be inspected if: 1) LA source materials are observed in adjacent areas and it appears to continue into the NUA, or 2) if the property owner provides information that indicates LA source materials may be present within a specific portion of the NUA. In this case, the area of concern within the NUA will be inspected as an SUA utilizing the guidelines outlined in Table 4-2.

#### 4.3.6.2 Soil Sampling

In general, three types of soil samples will be collected during DIs; characterization samples, re-characterization samples, and delineation samples. These types of soil samples are described below.

#### **Characterization Samples**

Previous investigation/screening activities focused only on high traffic areas of the property; therefore some portions of the property may not have been sampled. Characterization samples will be collected to characterize use areas that were not previously sampled.

**Re-Characterization Samples** 



Soil samples were collected during previous investigation/screening activities to determine the presence or absence of LA within soil. These samples were generally collected as 5-point composite samples from relatively large areas. Re-characterization samples will be collected from all previously sampled areas where previous sampling protocol was employed and results did not indicate LA (i.e., were non-detect).

#### **Delineation Samples**

Soil samples were collected during previous investigation/screening activities to determine the presence/absence of LA within soil, and were collected to characterize relatively large areas. Delineation samples will be collected to further define the extent and boundary of contamination. Delineation samples will be collected from areas where previous sample results indicate the presence of LA but where the sampled area exceeds the maximum area per DI soil sample as outlined in Table 4-2.

#### 4.3.6.2.1 Sample Collection

All DI soil samples will be collected in accordance with CDM-LIBBY-05 with the following exceptions:

- The maximum area that a single 30-point composite sample may include varies depending on the type of use area. Table 4-2 (Detailed Investigation) defines the maximum area per soil sample.
- Soil samples will not be collected in areas where vermiculite materials are observed. It should be noted that this approach is different than SI soil sampling procedures in areas where vermiculite was observed as discussed in Section 4.2.6.2.
- Samples collected outside the computed property boundary (as defined on the land survey) will be associated with the property assuming its ownership if the difference between assumed and computed property boundary is less than 10 feet. If the difference is greater than 10 feet, samples will be associated with the actual property address where the samples were collected.

#### 4.3.6.3 Exterior Inspection Documentation

An EPIF and VVEF will be completed for each property inspected as part of this SAP. EPIF's and VVEF's will be completed electronically as described in CDM-LIBBY-15.

Sample information and visual inspection results will be recorded on two separate property sketches. The property survey will be utilized as the baseline for these sketches. Sample information and visual inspection results may be combined on one sketch if quality and clarity can be maintained. Sketches will include the details indicated in Table 4-3.

Investigation teams will collect digital photographs in accordance with Section 4.5.6 of this SAP. Photographs will include access points, exterior hazards, pre-existing conditions, all areas where LA source materials are observed, and general interior photos.



In addition, investigation teams will review and provide feedback regarding GeoUnit-to-property relationships as discussed in the EPA Data Management Plan (EPA 2010).

## 4.4 Field QC Samples

Field QC samples are currently not required by EPA for bulk materials due to the homogenous nature of the material being sampled.

Field QC samples associated with soil samples are field duplicate samples. These samples are discussed in this section and summarized in Table 4-4.

Field duplicate samples will be collected at a rate of 1 per 20 field samples collected per sample type (i.e., 1 filed duplicate per 20 SI field samples, and 1 field duplicate per 20 DI field samples). Field duplicate samples will be collected from areas that are being sampled during one of the investigation activities discussed in the previous sections. However, individual composite points for the duplicate sample will be collected from different locations within the same use area than the original sample. Field duplicate samples will be collected in accordance with CDM-LIBBY-05. The investigation TL or FTL are responsible for maintaining overall GPI program soil field duplicate sample collection frequencies.

**Table 4-4 Summary of Field Quality Control Samples** 

Sample Type	Associated QC Sample	Collection Frequency	Analysis Frequency	Analysis Request	Acceptance Criteria
Soil	field duplicate	1 per 20 field samples	100%	PLM- VE/PLM-	<30% RPD
				Grav	

PLM-VE – polarized light microscopy visual area estimation method

PLM-Grav – polarized light microscopy gravimetric method

RPD – relative percent difference

N/A - not applicable

## 4.5 General Processes

This section describes the general field processes that will be used to support the sampling described in this SAP and includes references to the CDM SOPs and project-specific procedures when applicable.

## 4.5.1 Equipment Decontamination

Equipment used to collect, handle, or measure soil samples will be decontaminated before removing the equipment from any investigation site. Decontamination will be conducted in accordance with CDM SOP 4-5, Field Equipment Decontamination at Non-radioactive Sites (Appendix A) with the following modifications:

<u>Section 4.0, Required Equipment</u> - Plastic sheeting will not be used during decontamination procedures. ASTM Type II water will not be used. Rather, locally available de-ionized water will be used.



<u>Section 5.0, Procedures</u> - Decontamination water will not be captured and will be discharged to the ground at the worksite.

<u>Section 5.3, Sampling Equipment Decontamination</u> – Sampling equipment that has been decontaminated will not be wrapped in plastic sheeting or aluminum foil. As stated in CDM SOP 4-5, Section 5.0, all equipment will be decontaminated before and after use (i.e. rinse with locally available de-ionized water).

<u>Section 5.6, Waste Disposal</u> - Decontamination water will not be captured and will not be packaged, labeled, or stored as IDW. Decontamination water will be discharged to the ground at the worksite.

## 4.5.2 Investigation-Derived Waste

IDW at each property will consist of excess sample volume, spent decontamination supplies, and PPE. All IDW will be handled in accordance with CDM SOP 2-2, Guide to Handling IDW (Appendix A) with the following modification:

<u>Section 5.2, Offsite Disposal</u> – All IDW (not including excess soil volume) will be collected in transparent garbage bags and marked "IDW" with an indelible ink marker. These bags will be deposited into the asbestos contaminated waste stream for appropriate disposal at the local landfill. Excess soil volume will be returned to the use area from where it was collected.

### 4.5.3 Field Sample Data Sheets

Electronic FSDSs (eFSDSs) will be completed for each sample in accordance with CDM-LIBBY-15. The eFSDS is a record of specifics related to sample collection. eFSDSs are used to directly feed sample information into the project database and to connect analytical results to the sample collected. Examples of eFSDSs, which contain the required media-specific sample information for all samples collected under this SAP, are located in Appendix D.

## 4.5.4 Field Logbooks

Documentation of investigation field activities conducted under this SAP will be recorded in field logbooks maintained specifically for this sampling program. Logbooks are controlled documentation (i.e., sequentially numbered) and maintained by A&E administrative staff. Logbook numbers are cross-referenced on eFSDSs for efficient retrieval of information.

Detailed sampling notes will be recorded for each sample in accordance with CDM SOP 4-1, Field Logbook Content and Control. The logbook is an accounting of activities at the site and will duly note problems or deviations from the governing plans and observations relating to the sampling and analysis program. A new logbook page will be completed for each property visited. The header information should include the address, and the property owner's name. When closing out a logbook page with lineout and signature, the author will also print his/her name



underneath the signature. A&E administrative staff will email scanned copies of field logbooks, as they are completed to the A&E's project file coordinator. Original logbooks will be maintained in the A&E's office in Libby, Montana.

## 4.5.5 Sample Labeling and Identification

A unique alphanumeric code, or sample identifier (ID), will identify each sample collected during GPI sampling. The coding system will provide a tracking record to allow retrieval of information about a particular sample and to ensure that each sample is uniquely identified. Sample IDs will be sequential and not be representative of any particular building or equipment. Sample IDs will correlate with sample Location IDs, which will be identified on eFSDSs and in the field logbooks.

The sample labeling scheme is as follows:

2S-XXXXX (screening investigation)

2D-XXXXX (detailed investigation)

Where:

2S or 2D identifies that a sample is collected in accordance with this SAP and XXXXX represents a 5-digit numeric code

Preprinted adhesive sample labels will be signed out to sampling personnel by the A&E operations database manager. The labels are controlled to prevent duplication in assigning sample IDs. The labels will be affixed to both the inner and outer sample bags for soil samples. Sample labels will be used in accordance with CDM SOP 1-2, Sample Custody.

## 4.5.6 Photographic Documentation

Photographs will be taken with a digital camera at any place that field personnel determine necessary. Electronic photograph files will be saved each day to a project-designated computer housed at the A&E's Libby office and named so that photographs for a particular property or activity can easily be retrieved. The photograph file naming convention is as follows:

45 Montana Ave\_DI\_092110\_001

Where:

45 Montana Ave = the address where GPI activities occurred DI or SI = the specific activity being documented 092110 = the date the photo was taken 001 = the number of the photo taken at that property that day



Following completion of GPI activities, all photo files pertaining to a property will be copied to the A&E's server and also copied onto compact disc and filed in Libby along with other property-specific documentation.

### 4.5.7 Change Control

Logbook requirements are described in Section 4.5.4. Corrections to logbook entries require a single strikeout initial and date. The corrected information should be entered in close proximity to the existing entry. These procedures will also be followed for the correction of any hard copy field form (Appendix D). Electronically-captured data will be updated by field staff in accordance with the change control procedures contained in CDM-LIBBY-15. To the extent possible, field staff that originally collected the data will make the updates. Updated data will be submitted to EPA promptly in order to meet EPA reporting requirements.

All deviations from the guiding documents will be recorded in the logbooks by the investigation team and on the Libby Asbestos Project Record of Deviation/Request for Modification Form (Appendix F) by the property investigation field team leader.

#### 4.5.8 GPS Point Collection

GPS location coordinates will be collected in accordance with CDM-LIBBY-09. Coordinates will be collected for Location IDs. Coordinates for structures/buildings will be collected only if the building does not already have an assigned GPS location.

## 4.5.9 Field Sample Custody

Sample custody and documentation will follow the requirements specified in CDM SOP 1-2, Sample Custody (Appendix A) with the following clarifications:

#### 5.1 Transfer of Custody and Shipment -

- A COC record will not be completed in the field. Initial sample custody will be documented through the collection of sample information using eFSDS or hard copy FSDS, along with a physical sample.
- Sample labels/tags will be limited to a unique sample ID, which will be clearly indicated using pre-printed labels or hand-written on the ziptop sample bag for bulk samples, and both the inner and outer ziptop bag for soil samples.
- Investigation teams will securely place a custody seal on each individual sample.

All teams will ensure that samples, while in their possession, are maintained in a secure manner to prevent tampering, damage, or loss.

Whether sample information is collected electronically in the field or data-entered using hard copy FSDSs, investigation teams will follow the steps outlined in CDM-LIBBY-15 to synchronize sample information with the A&E's Libby Central Server.



Once data synchronization is complete, investigation teams can then relinquish samples directly to sample coordination staff or to a designated secure sample storage location. Once samples are received/retrieved, sample coordination staff will cross reference electronic sample IDs (from eFSDSs) with those on the physical samples, and will ultimately produce COC records and prepare the samples for transfer or shipping, as discussed in Sections 4.5.10 and 4.5.11.

## 4.5.10 Chain-of-Custody Records

For the Libby project, the COC record is employed as physical evidence of sample custody and condition from the sample coordination team to the receiving facility. A completed COC record is required to accompany each batch of samples, whether it is hand-delivered to the EPA laboratory coordinator (LC) or shipped to a processing or analytical facility.

The sample coordination team will produce COC records in accordance with CDM-LIBBY-15. Only quality-checked sample information will be used for COC records. In the event that electronic systems are unavailable (e.g., due to a power outage), hard copy COC records will be employed (Appendix E). Any hard copy COC records will be data-entered as soon as electronic systems are back online.

For hand-deliveries, a sample coordinator will relinquish samples and corresponding COC records to the EPA LC under strict custody. During relinquishment, the sample coordinator will complete the following information in the designated spaces at the bottom of the COC record: signature, company name, date, and time. The EPA LC will also complete the required information and will make a note regarding sample condition (e.g., OK – accept). The sample coordinator will retain the bottom copy of the COC record for the A&E's project record.

## 4.5.11 Sample Packaging and Shipping

Samples will be packaged and shipped in accordance with CDM SOP 2-1, Packaging and Shipping of Environmental Samples (Appendix A) with the following modifications:

<u>1.4 Required Equipment</u> – Vermiculite (or other absorbent material) or ice will not be used for packaging or shipping samples.

<u>1.5 Procedures</u> – No vermiculite or other absorbent material will be used to pack the samples. No ice will be used.

Samples will be hand-delivered to the EPA LC, picked up by a delivery service courier, or shipped by a delivery service to the designated facility or laboratory, as applicable. For hand-deliveries, the sample coordinator will package samples for transit such that they are contained and secure (i.e., will not be excessively jostled). Clean plastic totes with the lids secured or sample coolers may be used for this purpose.



For samples requiring shipment, prior to sealing the shipping container, the sample coordinator will complete the following information in the designated spaces at the bottom of the COC record: signature, company name, date, and time. The sample coordinator will retain the bottom copy of the COC record for the A&E's project record.

## 4.5.12 Field Equipment Maintenance

Field equipment maintenance will be conducted and documented in accordance with CDM SOP 5-1, Control of Measurement and Test Equipment (Appendix A).



## Section 5

## **Laboratory Operations**

EPA will be responsible for all sample analysis, including any sample processing prior to analysis. The A&E will be responsible for relinquishing all samples to the EPA LC, or processing facility or laboratory as designated by the EPA LC. The A&E sample coordinator will also be responsible for communicating with the EPA LC to relay pertinent sample and analysis information including sample quantities; special sample handling requirements, processing, or analysis concerns; and requested turnaround times.

This section discusses the analytical methods, custody and documentation procedures, quality assurance/quality control (QA/QC) requirements, and data management requirements to be employed by the laboratory in support of property investigation activities.

## 5.1 Analytical Methods and Turnaround Times

This section describes the analytical methods used for SI and DI samples.

An analytical summary sheet specific to sampling activities associated with this SAP will be distributed by EPA, and reviewed and approved by all participating laboratories prior to any sample handling. In order to clearly differentiate the samples collected for this investigation, each COC will reference the SAP-specific Summary of Preparation and Analytical Requirements for Asbestos in the comments section for each sample.

The A&E's sample coordinator will provide the EPA LC with requested turn-around times for all samples relinquished. In general, it is expected that analysis, including soil preparation, for all SI and DI soil and bulk samples will be complete within 10 (business) days and archived dust samples will be complete within 5 (business) days from the time the laboratory receives them.

## 5.1.1 PLM-VE/PLM-Grav – Soil Samples

Prior to analysis, all soil samples require a processing step. Soil samples will be processed using the current version of the Libby soil sample processing SOP (ISSI-LIBBY-01) (ISSI Consulting Group [ISSI] 2000) and the procedures included in the *Soil Preparation Work Plan* (TechLaw 2007). The A&E will indicate the current version of the soil sample processing SOP in the analysis request section of the COC record. It is the responsibility of the soil preparation facility to specify the appropriate PLM method as it corresponds to the specific sample fraction being submitted for analysis (i.e., fine ground or coarse fraction) on their COC records to the laboratory.



All soil samples collected as part of this effort, including field duplicate samples, will be analyzed for asbestos by the PLM visual estimation method (PLM-VE) and the PLM gravimetric method (PLM-Grav) in accordance with SOPs SRC-LIBBY-03 (Syracuse Research Corporation [SRC] 2003a) and SRC-LIBBY-01 (SRC 2002), respectively.

## 5.1.2 PLM-9002 - Bulk Material Samples

All bulk material samples collected as part of this effort will be analyzed by National Institute for Occupational Safety and Health (NIOSH) 9002, Issue 2, *Asbestos (bulk) by PLM* (NIOSH 1994), as specified on the COC record.

Because the level of detection is estimated (at less than 1 percent asbestos) for this method, no specific level of detection has been established for project samples analyzed using NIOSH 9002.

### 5.1.3 TEM – Dust Samples

Dust samples will not be collected as part of this SAP. However, archived dust samples collected as part of previous investigations (i.e., CSS) will be analyzed to support removal decision being determined as part of this SAP. The A&E investigation FTL will identify all archived dust samples that require analysis and communicate this to the A&E sample coordinator.

All archived CSS dust samples will be analyzed by transmission electron microscopy (TEM) in accordance with the project-amended ASTM D5755 method as described in SOP SRC-LIBBY-05 (SRC 2003b).

The laboratory will achieve the target method analytical sensitivity of 1,000 per square centimeter using direct preparation techniques. If necessary to achieve the target analytical sensitivity, indirect preparation techniques may be used as described in EPA-LIBBY-08 (EPA 2007).

## **5.2 Holding Times**

For the samples specified for collection in this SAP, no holding time requirements will be employed.

## **5.3 Laboratory Custody Procedures**

Laboratory custody procedures are provided in the QA management plans for each laboratory. These plans were independently audited and found to be satisfactory by EPA's laboratory audit team.

The basic laboratory sample custody process is as described herein. Upon receipt at the laboratory, each sample shipment will be inspected to assess the condition of the shipment and the individual samples. This inspection will include verifying sample integrity. The accompanying COC record will be cross-referenced with all of the samples in the shipment. The laboratory sample custodian will sign the COC record



and maintain a copy for their project files; the original COC record will be appended to the hard copy data report. Next, the sample custodian may assign a unique laboratory number to each sample on receipt. This number will identify the sample through all further handling at the laboratory. It is the laboratory's responsibility to maintain internal logbooks and records throughout sample preparation, analysis, data reporting, and sample archiving.

## 5.4 Laboratory QA/QC

The Libby Asbestos Project laboratory QA program may consist of laboratory certifications, team training and mentoring, analyst training, and laboratory audits. Laboratories that analyze field samples on the Libby project must maintain particular certifications and must satisfactorily complete project-specific training requirements to ensure that proper QA/QC practices are conducted during sample analysis.

Analytical laboratories will be provided a copy of and will adhere to the requirements of this SAP. Samples collected under this SAP will be analyzed in accordance with standard EPA and/or nationally-recognized analytical procedures (i.e., Good Laboratory Practices) in order to provide analytical data of known quality and consistency.

## 5.5 Laboratory Documentation and Reporting

All deviations from project-specific and method analytical guidance documents, or this SAP, will be recorded on the Libby Asbestos Project Laboratory Record of Modification Form (Appendix F). Any deviations that impact, or have the potential to impact, investigation objectives will be discussed with the OU4 EPA RPM and A&E FTL prior to implementation. In addition, the Record of Modification Form will be used to document any information of interest as requested by EPA. As modifications are approved by EPA and implemented, the EPA LC will communicate the changes to the EPA laboratories.

Sample results data will be delivered to the EPA in accordance with the current version of the EPA Data Management Plan (EPA 2010).

## 5.6 Laboratory Nonconformance

Laboratories will immediately notify the EPA LC if major problems occur (e.g., catastrophic equipment failure). The EPA LC will then notify the A&E sample coordinator of potential impacts to turn-around times. Other nonconformance issues, such as those found during performance evaluations or audits, will be addressed on a case-by-case basis by the EPA's laboratory audit team.



## Section 6

## **Assessments and Oversight**

Assessments and oversight reports to management are necessary to ensure that procedures are followed as required and that deviations from procedures are documented. These reports also serve to keep management current on field activities. Assessment, oversight reports, and response actions are discussed below.

#### 6.1 Assessments

Performance assessments are quantitative checks on the quality of a measurement system and are appropriate to analytical work. Performance assessments for the laboratories may be accomplished by submitting blind reference material (i.e., performance evaluation samples). These assessment samples are samples with known concentrations that are submitted to the laboratories without identifying them as such to the laboratories. Performance assessments will be coordinated by EPA.

System assessments are qualitative reviews of different aspects of project work to check the use of appropriate QC measures and the general function of the QA system. Field and office system assessments will be performed under the direction of the A&E's QA director, with support from the A&E's project QA coordinator. Quality Procedure 6.2, as defined in the A&E's QA Manual (CDM 2007), defines requirements for conducting field and office system assessments. Due to the level of effort for sampling and the duration of the activities discussed in this SAP, both a field audit and an office audit are scheduled for GPIs annually. Laboratory system assessments/audits will be coordinated by EPA.

## **6.2** Corrective Actions

Corrective actions will be implemented on a case-by-case basis to address quality problems. Minor actions taken in the field to immediately correct a quality problem will be documented in the applicable field logbook and a verbal report will be provided to the A&E's project manager and/or site manager. Major corrective actions taken in the field will be approved by the OU4 EPA remedial project manager and A&E's project manager prior to implementation of the change. Major response actions are those that may affect the quality or objective of the investigation. Quality problems that cannot be corrected quickly through routine field procedures may require implementation of a corrective action request (CAR) form, as provided in the A&E's QA Manual (CDM 2007b).

All CARs will be submitted to either the A&E's QA Director or project QA coordinator for review and issuance. The A&E's project manager or project QA coordinator will notify their QA director when quality problems arise that may require a CAR. CAR forms will be completed according to Quality Procedure 8.1 of the A&E's QA Manual (CDM 2007b).



In addition, when modifications to this SAP are required, either for field or laboratory activities, a Libby Asbestos Project Record of Modification Form (Appendix E) must be completed.

## 6.3 Reports to Management

QA reports will be provided to management for routine audits and whenever quality problems are encountered. Field staff will note any quality problems on eFSDSs or in field log notes. Further, the A&E's project manager will inform the project QA coordinator upon encountering quality issues that cannot be immediately corrected. Weekly reports and change request forms are not required for work performed under this SAP.



# Section 7 Data Review and Verification

Laboratory results will be reviewed and verified for compliance with project reporting requirements. Data review and verification, and DQO reconciliation are discussed in Sections 7.1 and 7.2, respectively.

## 7.1 Data Review and Verification Requirements

Data review (i.e., QC review) to be performed by designated A&E staff includes cross-checking that sample IDs and sample dates have been reported correctly on the preliminary laboratory report, and that calculated analytical sensitivities or detection levels are as expected. EPA will be responsible for reviewing all analytical data deliverables prepared for samples collected as part of this SAP, and coordinating with the laboratory for corrections and re-issuances of data reports.

Data verification includes checking that results have been transferred correctly from laboratory data printouts to the finalized laboratory report and to the electronic data deliverable (EDD), and that both the laboratory report and EDD are complete before they are submitted to EPA. This function is performed primarily as a function of built-in QC checks in the project database (managed by EPA) when data is uploaded. As a result, data users may be the first personnel to encounter discrepancies. If discrepancies are found, the data user will contact EPA, who will then notify the appropriate laboratory in order to correct the issue.

## 7.2 DQO Reconciliation

The DQOs presented in Section 3 will be reconciled during the data review process. During this process, the A&E's GPI team members will compare the reported results against the project-specific action levels discussed in the DQOs. Attainment of project DQOs results in determining what areas do or do not contain LA and/or LA source materials for development of property-specific removal action work plans. Non-attainment of project DQOs may result in additional follow up visits to the property for additional sample collection or field observations in order to achieve DQOs.



# Section 8 References

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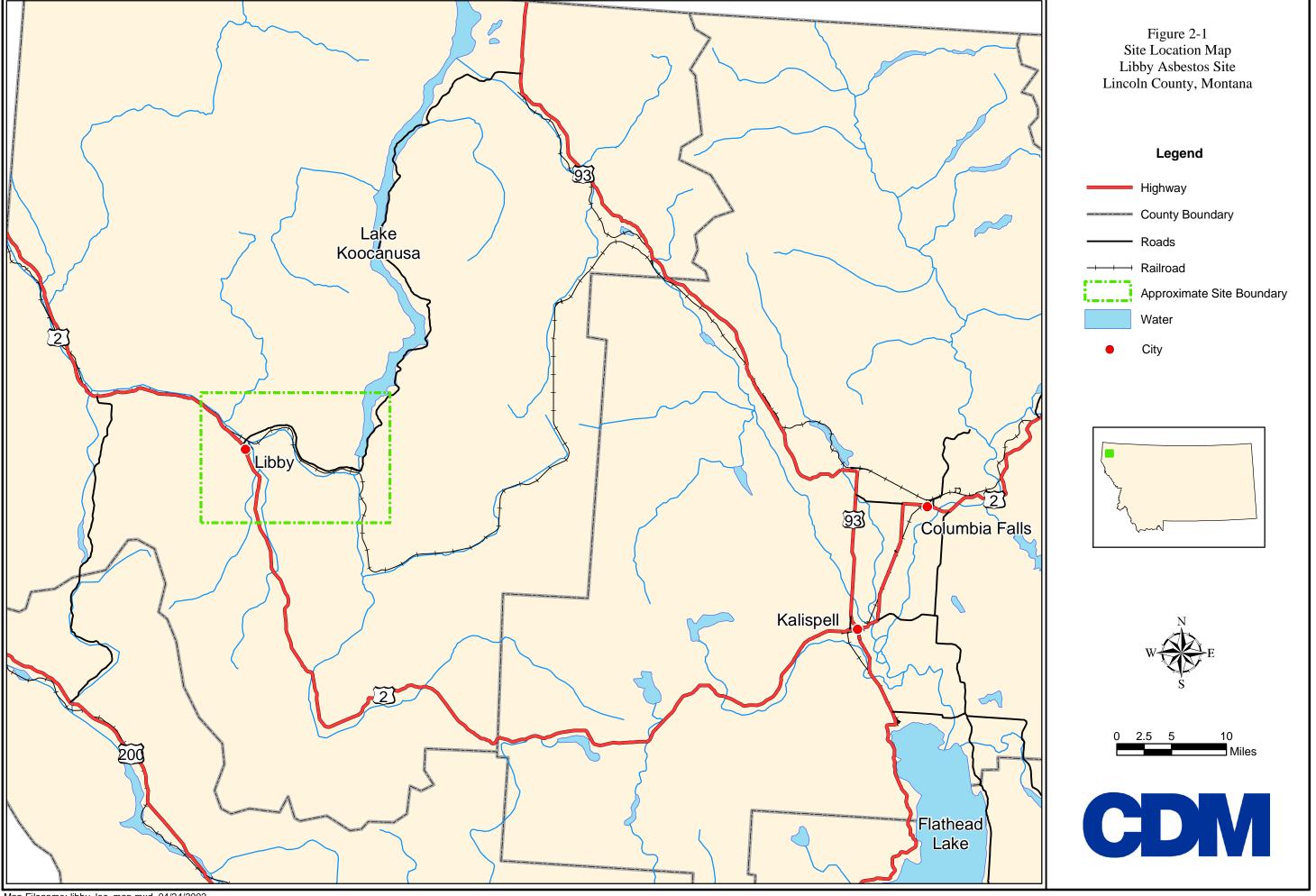
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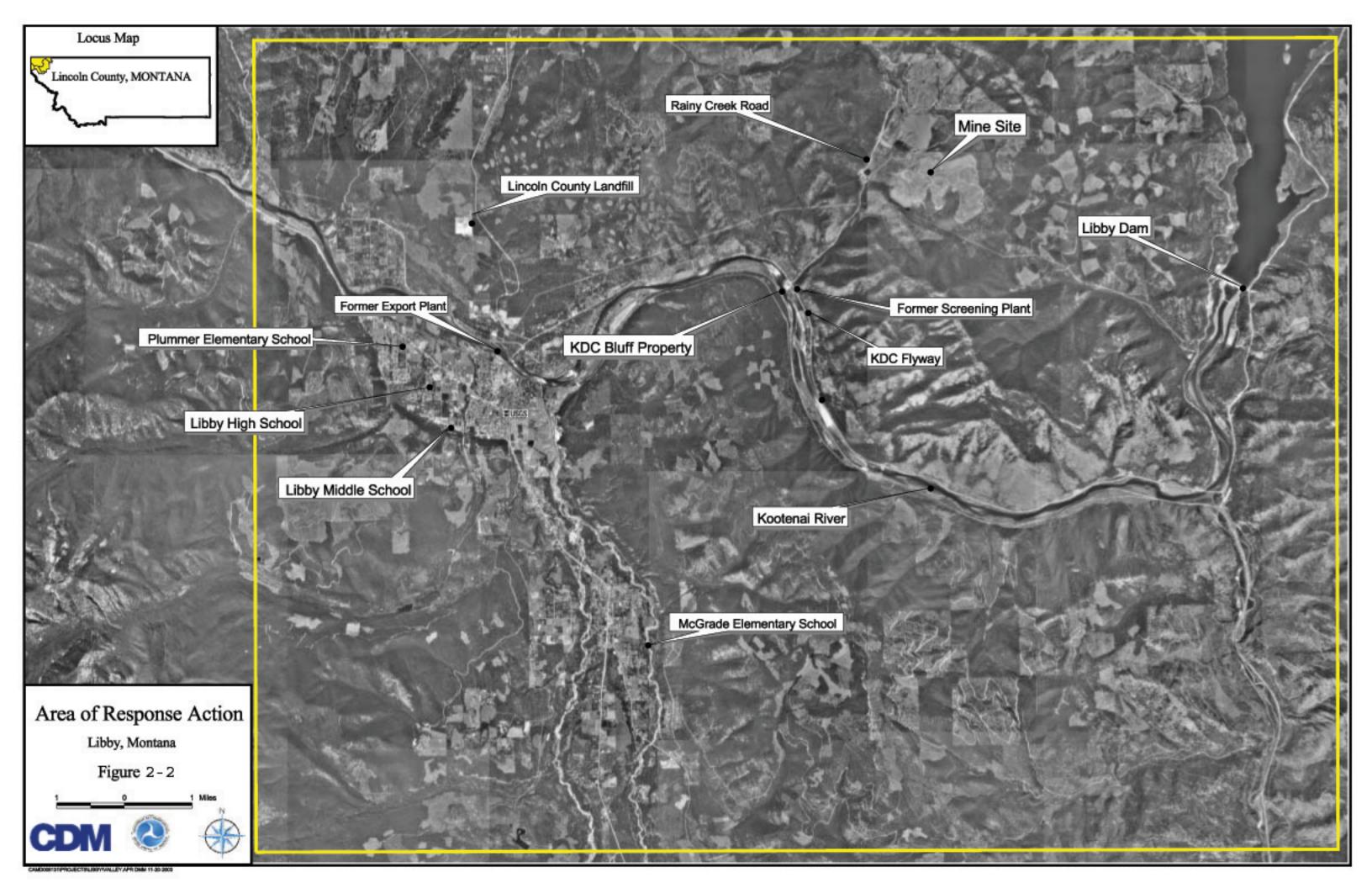
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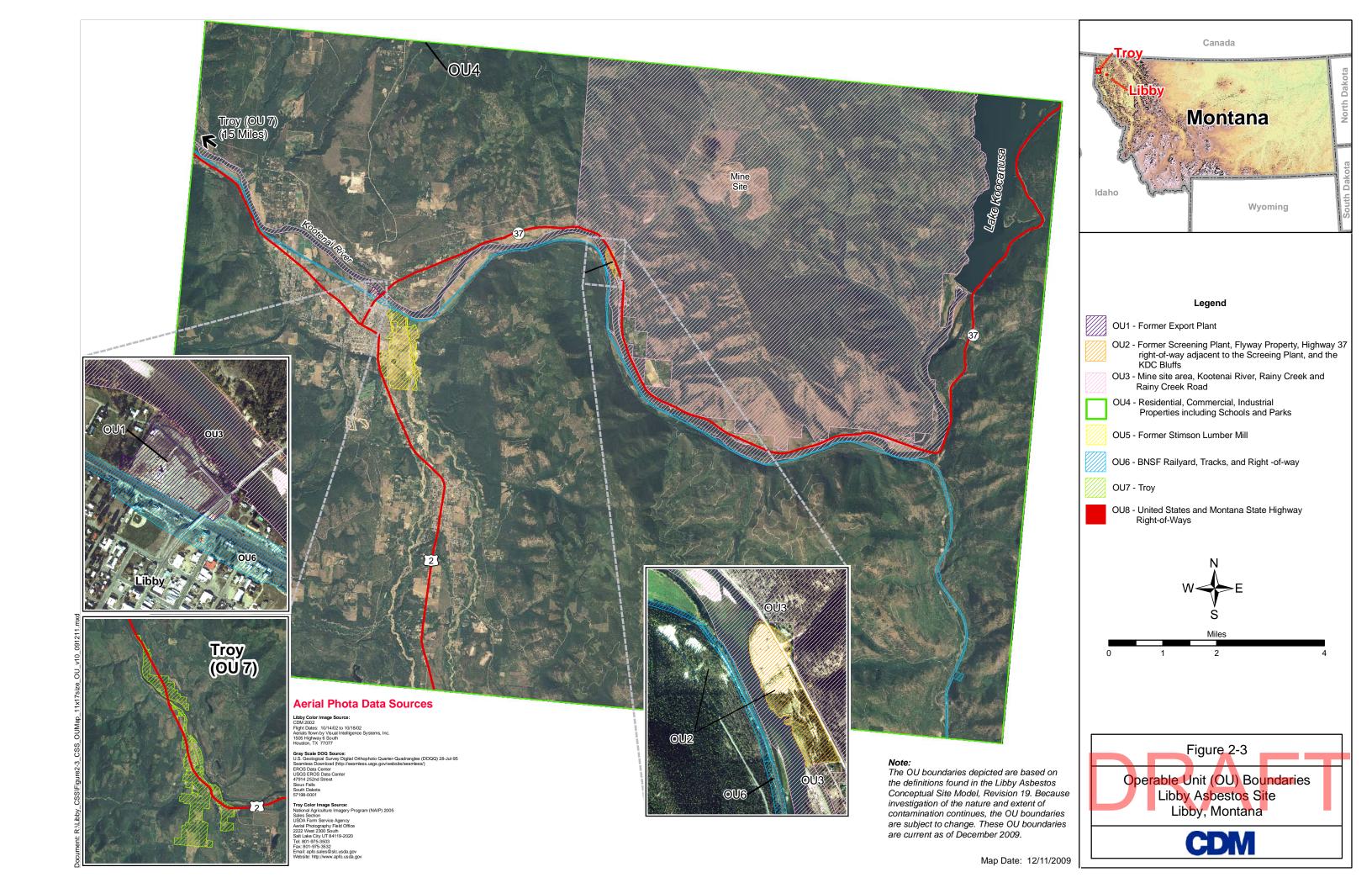
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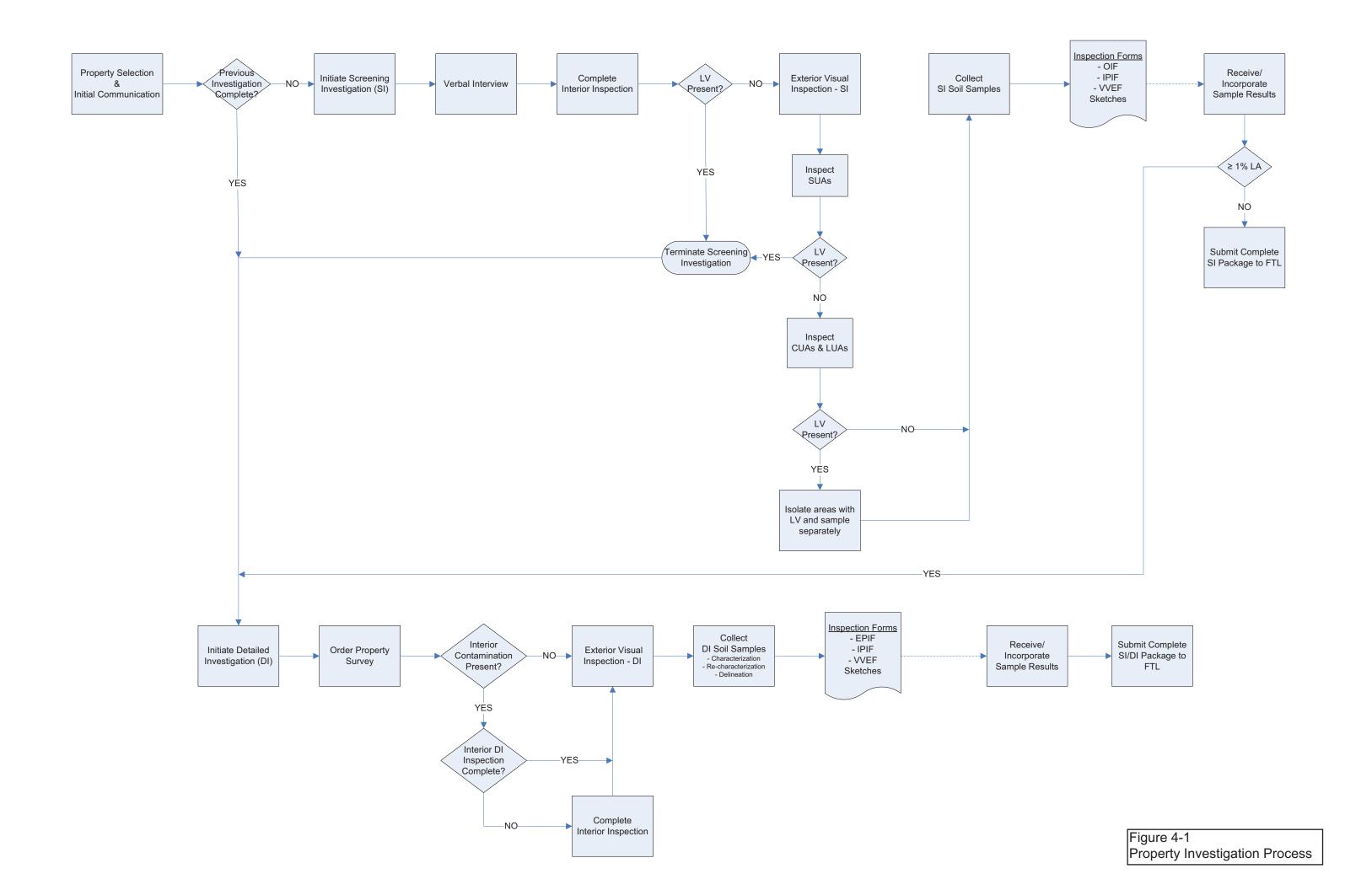
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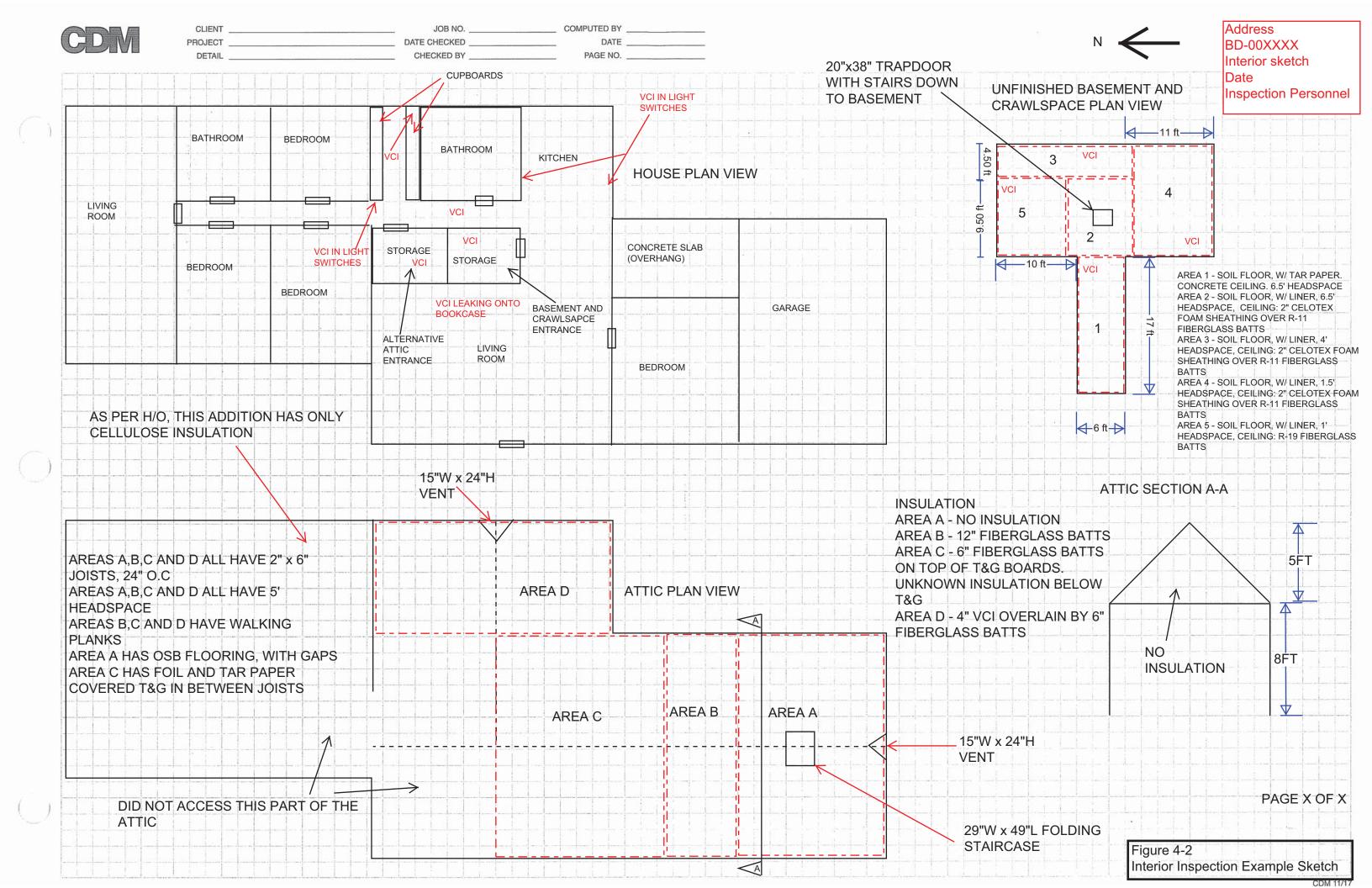


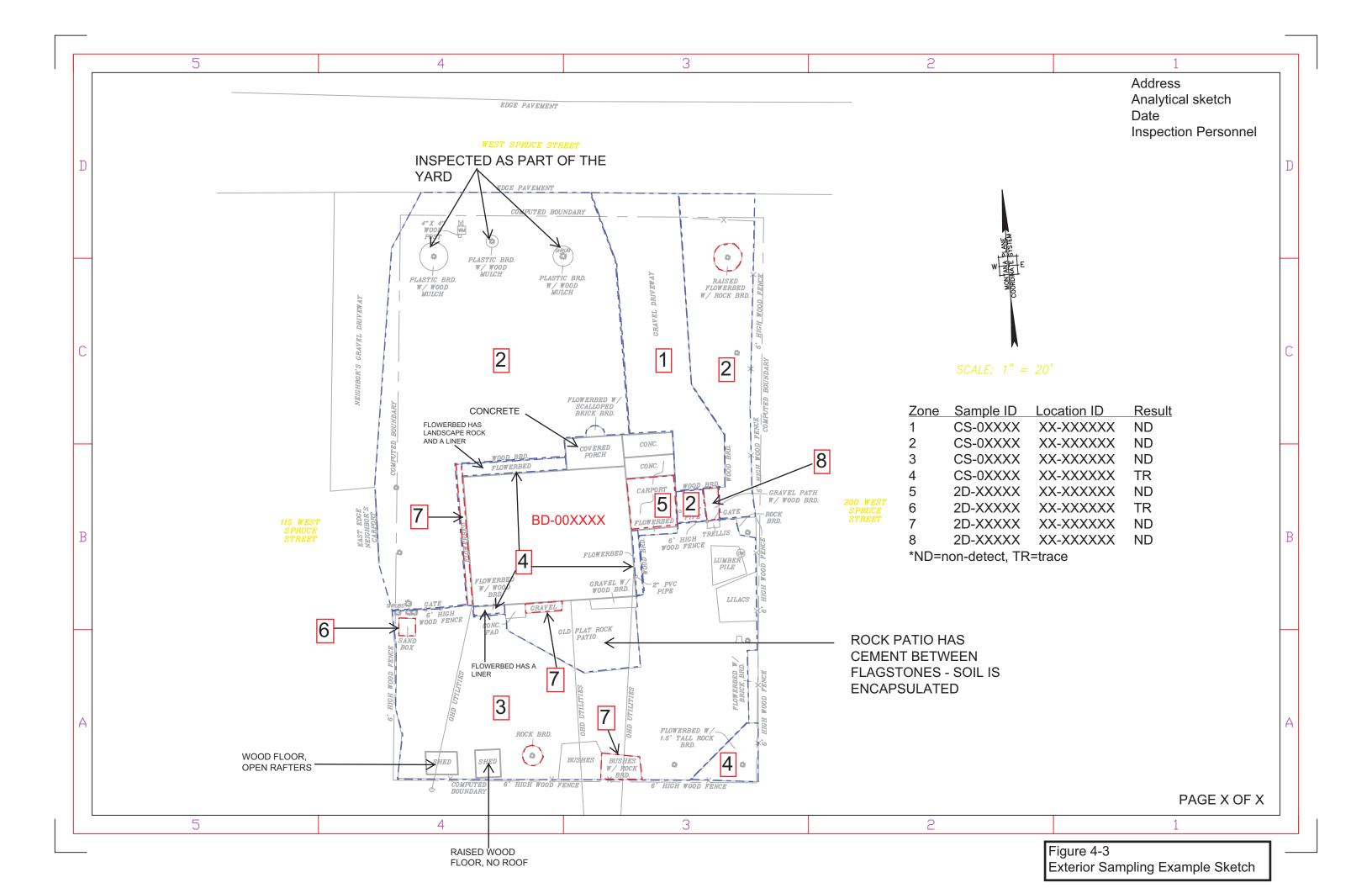


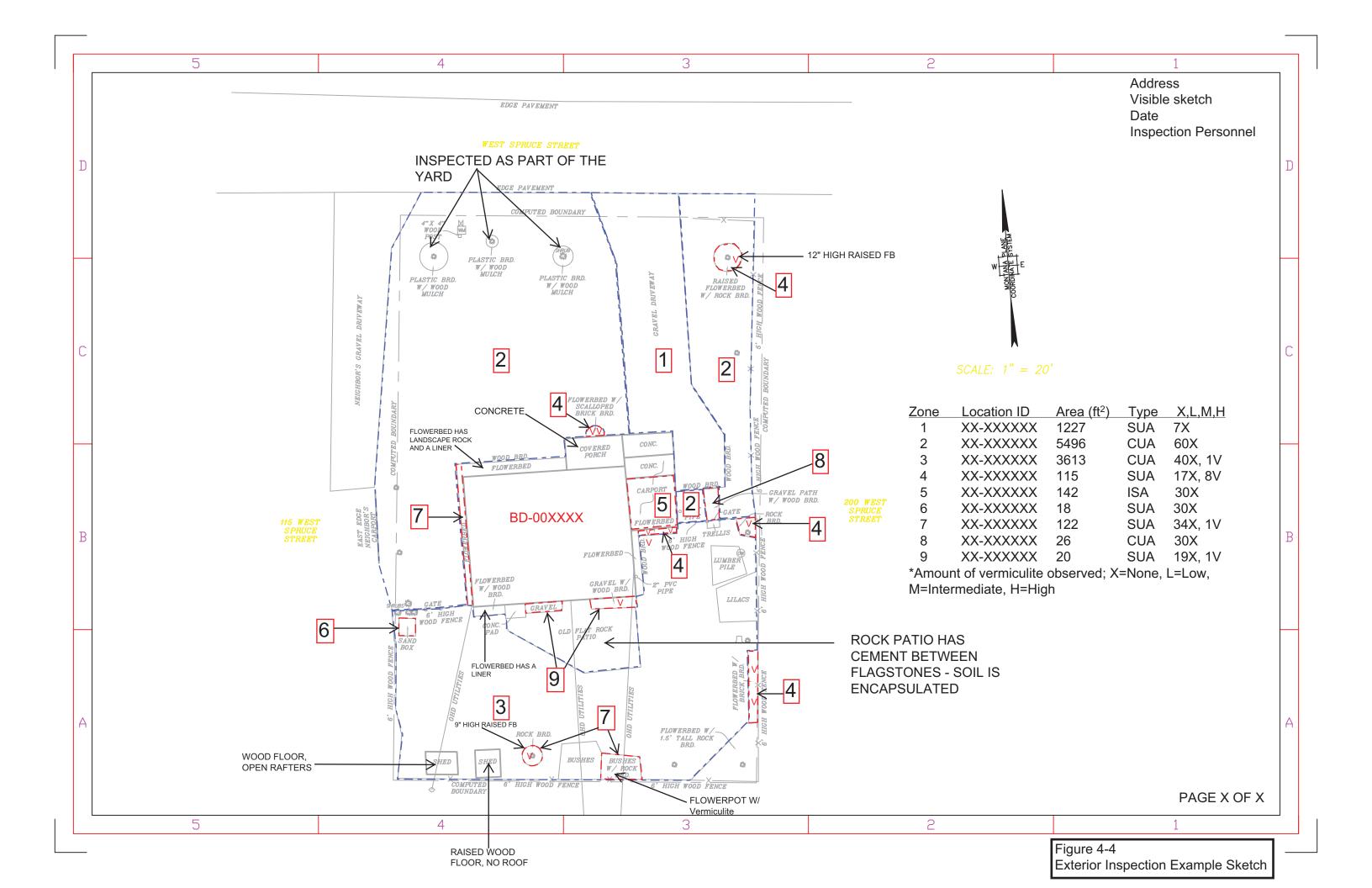












# **APPENDIX A Standard Operating Procedures**

#### **National Guidelines**

Appendix A to Subpart E of Part 763 – Interim Transmission Electron Microscopy Analytical Methods – Mandatory and Nonmandatory – and Mandatory Section to Determine Completion of Response Actions Section 763.86 to Part E of Subpart 763 – Sampling, Asbestos Containing Materials in Schools

#### **CDM Standard Operating Procedures**

Sample Custody (CDM SOP 1-2)

Packaging and Shipping of Environmental Samples (CDM SOP 2-1)

Guide to Handling of Investigation-Derived Waste (CDM SOP 2-2)

Field Logbook Content and Control (CDM SOP 4-1)

Field Equipment Decontamination at Non-radioactive Sites (CDM SOP 4-5)

Control of Measurement and Test Equipment (CDM SOP 5-1)

#### **Project-specific Standard Operating Procedures**

Project-specific Standard Operating Procedure for 30-point Composite Soil Sample Collection (CDM-LIBBY-05)

Project-specific Standard Operating Procedure for Visual Vermiculite Soil Inspections (CDM-LIBBY-06)

Project-specific Standard Operating Procedure for Global Positioning System Coordinate Collection and Handling (CDM-LIBBY-09)

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#### § 763.86 Sampling.

- (a) Surfacing material. An accredited inspector shall collect, in a statistically random manner that is representative of the homogeneous area, bulk samples from each homogeneous area of friable surfacing material that is not assumed to be ACM, and shall collect the samples as follows:
- (1) At least three bulk samples shall be collected from each homogeneous area that is 1,000 ft 2 or less, except as provided in § 763.87(c)(2).
- (2) At least five bulk samples shall be collected from each homogeneous area that is greater than 1,000 ft 2 but less than or equal to 5,000 ft 2, except as provided in § 763.87(c)(2).
- (3) At least seven bulk samples shall be collected from each homogeneous area that is greater than 5,000 ft 2, except as provided in § 763.87(c)(2).
- (b) Thermal system insulation. (1) Except as provided in paragraphs (b) (2) through (4) of this section and § 763.87(c), an accredited inspector shall collect, in a randomly distributed manner, at least three bulk samples from each homogeneous area of thermal system insulation that is not assumed to be ACM.
- (2) Collect at least one bulk sample from each homogeneous area of patched thermal system insulation that is not assumed to be ACM if the patched section is less than 6 linear or square feet.
- (3) In a manner sufficient to determine whether the material is ACM or not ACM, collect bulk samples from each insulated mechanical system that is not assumed to be ACM where cement or plaster is used on fittings such as tees, elbows, or valves, except as provided under § 763.87(c)(2).
- (4) Bulk samples are not required to be collected from any homogeneous area where the accredited inspector has determined that the thermal system insulation is fiberglass, foam glass, rubber, or other non-ACBM.
- (c) Miscellaneous material. In a manner sufficient to determine whether material is ACM or not ACM, an accredited inspector shall collect bulk samples from each homogeneous area of friable miscellaneous material that is not assumed to be ACM.
- (d) Nonfriable suspected ACBM. If any homogeneous area of nonfriable suspected ACBM is not assumed to be ACM, then an accredited inspector shall collect, in a manner sufficient to determine whether the material is ACM or not ACM, bulk samples from the homogeneous area of nonfriable suspected ACBM that is not assumed to be ACM.



## Title 40 -- Protection of Environment

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Appendix A to Subpart E of Part 763 -- Interim Transmission Electron Microscopy Analytical Methods -- Mandatory and Nonmandatory -- and Mandatory Section to Determine Completion of Response Actions

#### I. Introduction

The following appendix contains three units. The first unit is the mandatory transmission electron microscopy (TEM) method which all laboratories must follow; it is the minimum requirement for analysis of air samples for asbestos by TEM. The mandatory method contains the essential elements of the TEM method. The second unit contains the complete non-mandatory method. The non-mandatory method supplements the mandatory method by including additional steps to improve the analysis. EPA recommends that the non-mandatory method be employed for analyzing air filters; however, the laboratory may choose to employ the mandatory method. The non-mandatory method contains the same minimum requirements as are outlined in the mandatory method. Hence, laboratories may choose either of the two methods for analyzing air samples by TEM.

The final unit of this Appendix A to subpart E defines the steps which must be taken to determine completion of response actions. This unit is mandatory.

#### II. Mandatory Transmission Electron Microscopy Method

#### A. Definitions of Terms

- 1. Analytical sensitivity -- Airborne asbestos concentration represented by each fiber counted under the electron microscope. It is determined by the air volume collected and the proportion of the filter examined. This method requires that the analytical sensitivity be no greater than 0.005 structures/cm <sup>3</sup>.
- 2. *Asbestiform* -- A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.
- 3. Aspect ratio -- A ratio of the length to the width of a particle. Minimum aspect ratio as defined by this method is equal to or greater than 5:1.
- 4. *Bundle* -- A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.
- 5. Clean area -- A controlled environment which is maintained and monitored to assure a low probability of asbestos contamination to materials in that space. Clean areas used in this method have HEPA filtered air under positive pressure and are capable of sustained operation with an open laboratory blank which on subsequent analysis has an average of less than 18 structures/mm <sup>2</sup> in an area of 0.057 mm <sup>2</sup> (nominally 10 200-mesh grid openings) and a maximum of 53 structures/mm <sup>2</sup> for any single preparation for that same area.
- 6. *Cluster* -- A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two

intersections.

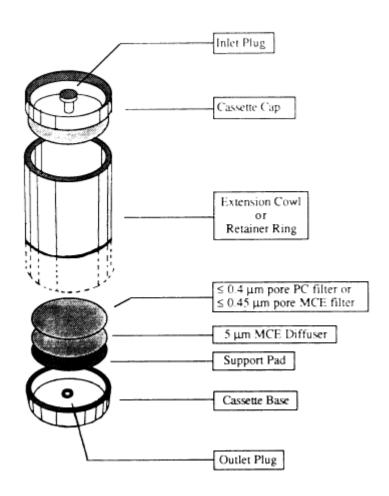
- 7. ED -- Electron diffraction.
- 8. EDXA -- Energy dispersive X-ray analysis.
- 9. Fiber -- A structure greater than or equal to 0.5 µm in length with an aspect ratio (length to width) of 5:1 or greater and having substantially parallel sides.
- 10. *Grid* -- An open structure for mounting on the sample to aid in its examination in the TEM. The term is used here to denote a 200-mesh copper lattice approximately 3 mm in diameter.
- 11. *Intersection* -- Nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater.
- 12. *Laboratory sample coordinator* -- That person responsible for the conduct of sample handling and the certification of the testing procedures.
- 13. Filter background level -- The concentration of structures per square millimeter of filter that is considered indistinguishable from the concentration measured on a blank (filters through which no air has been drawn). For this method the filter background level is defined as 70 structures/mm <sup>2</sup>.
- 14. *Matrix* -- Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.
- 15. NSD -- No structure detected.
- 16. Operator -- A person responsible for the TEM instrumental analysis of the sample.
- 17. *PCM* -- Phase contrast microscopy.
- 18. SAED -- Selected area electron diffraction.
- 19. SEM -- Scanning electron microscope.
- 20. STEM -- Scanning transmission electron microscope.
- 21. Structure -- a microscopic bundle, cluster, fiber, or matrix which may contain asbestos.
- 22. S/cm 3 -- Structures per cubic centimeter.
- 23. *S/mm 2* -- Structures per square millimeter.
- 24. *TEM* -- Transmission electron microscope.

#### **B.** Sampling

- 1. The sampling agency must have written quality control procedures and documents which verify compliance.
- 2. Sampling operations must be performed by qualified individuals completely independent of the abatement contractor to avoid possible conflict of interest (References 1, 2, 3, and 5 of Unit II.J.).
- 3. Sampling for airborne asbestos following an abatement action must use commercially available cassettes.

- 4. Prescreen the loaded cassette collection filters to assure that they do not contain concentrations of asbestos which may interfere with the analysis of the sample. A filter blank average of less than 18 s/mm $^2$  in an area of 0.057 mm $^2$  (nominally 10 200-mesh grid openings) and a single preparation with a maximum of 53 s/mm $^2$  for that same area is acceptable for this method.
- 5. Use sample collection filters which are either polycarbonate having a pore size less than or equal to  $0.4 \mu m$  or mixed cellulose ester having a pore size less than or equal to  $0.45 \mu m$ .
- 6. Place these filters in series with a 5.0  $\mu$ m backup filter (to serve as a diffuser) and a support pad. See the following Figure 1:

FIGURE I -- SAMPLING CASSETTE CONFIGURATION

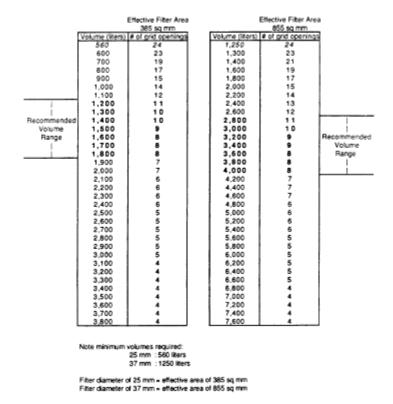


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- 7. Reloading of used cassettes is not permitted.
- 8. Orient the cassette downward at approximately 45 degrees from the horizontal.
- 9. Maintain a log of all pertinent sampling information.
- 10. Calibrate sampling pumps and their flow indicators over the range of their intended use with a recognized standard. Assemble the sampling system with a representative filter (not the filter which will be used in sampling) before and after the sampling operation.
- 11. Record all calibration information.

- 12. Ensure that the mechanical vibrations from the pump will be minimized to prevent transferral of vibration to the cassette.
- 13. Ensure that a continuous smooth flow of negative pressure is delivered by the pump by damping out any pump action fluctuations if necessary.
- 14. The final plastic barrier around the abatement area remains in place for the sampling period.
- 15. After the area has passed a thorough visual inspection, use aggressive sampling conditions to dislodge any remaining dust. (See suggested protocol in Unit III.B.7.d.)
- 16. Select an appropriate flow rate equal to or greater than 1 liter per minute (L/min) or less than 10 L/min for 25 mm cassettes. Larger filters may be operated at proportionally higher flow rates.
- 17. A minimum of 13 samples are to be collected for each testing site consisting of the following:
- a. A minimum of five samples per abatement area.
- b. A minimum of five samples per ambient area positioned at locations representative of the air entering the abatement site.
- c. Two field blanks are to be taken by removing the cap for not more than 30 seconds and replacing it at the time of sampling before sampling is initiated at the following places:
- i. Near the entrance to each abatement area.
- ii. At one of the ambient sites. (DO NOT leave the field blanks open during the sampling period.)
- d. A sealed blank is to be carried with each sample set. This representative cassette is not to be opened in the field.
- 18. Perform a leak check of the sampling system at each indoor and outdoor sampling site by activating the pump with the closed sampling cassette in line. Any flow indicates a leak which must be eliminated before initiating the sampling operation.
- 19. The following Table I specifies volume ranges to be used:

TABLE 1--NUMBER OF 200 MESH EM GRID OPENINGS (0.0057 MM<sup>2</sup>) THAT NEED TO BE ANALYZED TO MAINTAIN SENSITIVITY OF 0.005 STRUCTURES/CC BASED ON VOLUME AND EFFECTIVE FILTER AREA



- 20. Ensure that the sampler is turned upright before interrupting the pump flow.
- 21. Check that all samples are clearly labeled and that all pertinent information has been enclosed before transfer of the samples to the laboratory.
- 22. Ensure that the samples are stored in a secure and representative location.
- 23. Do not change containers if portions of these filters are taken for other purposes.
- 24. A summary of Sample Data Quality Objectives is shown in the following Table II:

TABLE II--SUMMARY OF SAMPLING AGENCY DATA QUALITY OBJECTIVES

This table summarizes the data quality objectives from the performance of this method in terms of precision, accuracy, complications, representativeness, and comparability. These objectives are assured by the periodic control checks and reference checks itself here and described in the text of the method.

Unit Operation	OC Check	Frequency	Conformance Expectation
Sampling materials	Scaled blank	1 per I/O site	95%
Sample procedures	Field blanks	2 per I/O site	95%
	Pump calibration	Before and after each field series	90%
Sample custody	Review of chain-of-custody record	Each sample	95% complete
Sample shipment	Review of sending report	Each sample	95% correlate

#### C. Sample Shipment

Ship bulk samples to the analytical laboratory in a separate container from air samples.

#### D. Sample Receiving

1. Designate one individual as sample coordinator at the laboratory. While that individual will normally be available to receive samples, the coordinator may train and supervise others in receiving procedures for those times when he/she is not available.

2. Bulk samples and air samples delivered to the analytical laboratory in the same container shall be rejected.

#### E. Sample Preparation

- 1. All sample preparation and analysis shall be performed by a laboratory independent of the abatement contractor.
- 2. Wet-wipe the exterior of the cassettes to minimize contamination possibilities before taking them into the clean room facility.
- 3. Perform sample preparation in a well-equipped clean facility.
- >Note: The clean area is required to have the following minimum characteristics. The area or hood must be capable of maintaining a positive pressure with make-up air being HEPA-filtered. The cumulative analytical blank concentration must average less than 18 s/mm $^2$  in an area of 0.057 mm $^2$  (nominally 10 200-mesh grid openings) and a single preparation with a maximum of 53 s/mm $^2$  for that same area.
- 4. Preparation areas for air samples must not only be separated from preparation areas for bulk samples, but they must be prepared in separate rooms.
- 5. Direct preparation techniques are required. The object is to produce an intact film containing the particulates of the filter surface which is sufficiently clear for TEM analysis.
- a. TEM Grid Opening Area measurement must be done as follows:
- i. The filter portion being used for sample preparation must have the surface collapsed using an acetone vapor technique.
- ii. Measure 20 grid openings on each of 20 random 200-mesh copper grids by placing a grid on a glass and examining it under the PCM. Use a calibrated graticule to measure the average field diameters. From the data, calculate the field area for an average grid opening.
- iii. Measurements can also be made on the TEM at a properly calibrated low magnification or on an optical microscope at a magnification of approximately 400X by using an eyepiece fitted with a scale that has been calibrated against a stage micrometer. Optical microscopy utilizing manual or automated procedures may be used providing instrument calibration can be verified.
- b. TEM specimen preparation from polycarbonate (PC) filters. Procedures as described in Unit III.G. or other equivalent methods may be used.
- c. TEM specimen preparation from mixed cellulose ester (MCE) filters.
- i. Filter portion being used for sample preparation must have the surface collapsed using an acetone vapor technique or the Burdette procedure (Ref. 7 of Unit II.J.)
- ii. Plasma etching of the collapsed filter is required. The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher. Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the conditions that should be used. Insufficient etching will result in a failure to expose embedded filters, and too much etching may result in loss of particulate from the surface. As an interim measure, it is recommended that the time for ashing of a known weight of a collapsed filter be established and that the etching rate be calculated in terms of micrometers per second. The actual etching time used for the particulate asher and operating conditions will then be set such that a 1-2  $\mu$ m (10 percent) layer of collapsed surface will be removed.

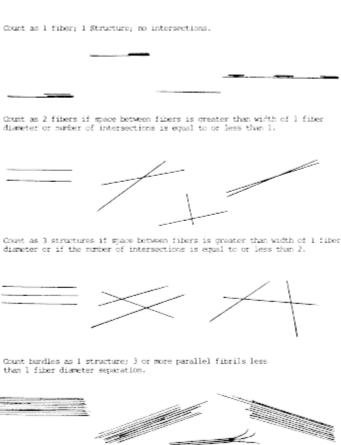
iii. Procedures as described in Unit III. or other equivalent methods may be used to prepare samples.

#### F. TEM Method

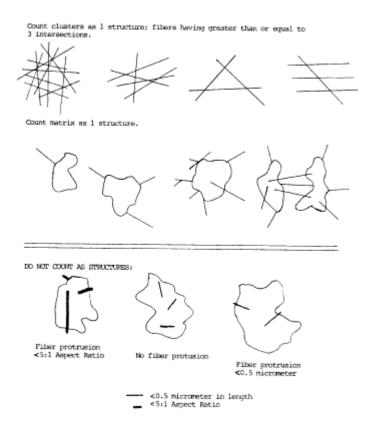
- 1. An 80-120 kV TEM capable of performing electron diffraction with a fluorescent screen inscribed with calibrated gradations is required. If the TEM is equipped with EDXA it must either have a STEM attachment or be capable of producing a spot less than 250 nm in diameter at crossover. The microscope shall be calibrated routinely for magnification and camera constant.
- 2. Determination of Camera Constant and ED Pattern Analysis. The camera length of the TEM in ED operating mode must be calibrated before ED patterns on unknown samples are observed. This can be achieved by using a carbon-coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thicker gold film would normally give multiple gold rings, but it will tend to mask weaker diffraction spots from the unknown fibrous particulate. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings are unnecessary on zone-axis ED patterns. An average camera constant using multiple gold rings can be determined. The camera constant is one-half the diameter of the rings times the interplanar spacing of the ring being measured.
- 3. Magnification Calibration. The magnification calibration must be done at the fluorescent screen. The TEM must be calibrated at the grid opening magnification (if used) and also at the magnification used for fiber counting. This is performed with a cross grating replica (e.g., one containing 2,160 lines/mm). Define a field of view on the fluorescent screen either by markings or physical boundaries. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric). A logbook must be maintained, and the dates of calibration and the values obtained must be recorded. The frequency of calibration depends on the past history of the particular microscope. After any maintenance of the microscope that involved adjustment of the power supplied to the lenses or the high-voltage system or the mechanical disassembly of the electron optical column apart from filament exchange, the magnification must be recalibrated. Before the TEM calibration is performed, the analyst must ensure that the cross grating replica is placed at the same distance from the objective lens as the specimens are. For instruments that incorporate a eucentric tilting specimen stage, all specimens and the cross grating replica must be placed at the eucentric position.
- 4. While not required on every microscope in the laboratory, the laboratory must have either one microscope equipped with energy dispersive X-ray analysis or access to an equivalent system on a TEM in another laboratory.
- 5. Microscope settings: 80-120 kV, grid assessment 250-1,000X, then 15,000-20,000X screen magnification for analysis.
- 6. Approximately one-half (0.5) of the predetermined sample area to be analyzed shall be performed on one sample grid preparation and the remaining half on a second sample grid preparation.
- 7. Individual grid openings with greater than 5 percent openings (holes) or covered with greater than 25 percent particulate matter or obviously having nonuniform loading must not be analyzed.
- 8. Reject the grid if:
- a. Less than 50 percent of the grid openings covered by the replica are intact.
- b. The replica is doubled or folded.

- c. The replica is too dark because of incomplete dissolution of the filter.
- 9. Recording Rules.
- a. Any continuous grouping of particles in which an asbestos fiber with an aspect ratio greater than or equal to 5:1 and a length greater than or equal to  $0.5~\mu m$  is detected shall be recorded on the count sheet. These will be designated asbestos structures and will be classified as fibers, bundles, clusters, or matrices. Record as individual fibers any contiguous grouping having 0, 1, 0 or 2 definable intersections. Groupings having more than 2 intersections are to be described as cluster or matrix. An intersection is a nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater. See the following Figure 2:

FIGURE 2--COUNTING GUIDELINES USED IN DETERMINING ASBESTOS STRUCTURES



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- i. Fiber. A structure having a minimum length greater than or equal to  $0.5~\mu m$  and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.
- ii. *Bundle*. A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.
- iii. *Cluster.* A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.
- iv. *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.
- b. Separate categories will be maintained for fibers less than 5  $\mu m$  and for fibers equal to or greater than 5  $\mu m$  in length.
- c. Record NSD when no structures are detected in the field.
- d. Visual identification of electron diffraction (ED) patterns is required for each asbestos structure counted which would cause the analysis to exceed the 70 s/mm <sup>2</sup> concentration. (Generally this means the first four fibers identified as asbestos must exhibit an identifiable diffraction pattern for chrysotile or amphibole.)
- e. The micrograph number of the recorded diffraction patterns must be reported to the client and maintained in the laboratory's quality assurance records. In the event that examination of the pattern by a qualified individual indicates that the pattern has been misidentified visually, the client shall be contacted.
- f. Energy Dispersive X-ray Analysis (EDXA) is required of all amphiboles which would cause the analysis results to exceed the 70 s/mm <sup>2</sup> concentration. (Generally speaking, the first 4

amphiboles would require EDXA.)

- g. If the number of fibers in the nonasbestos class would cause the analysis to exceed the 70 s/mm $^2$  concentration, the fact that they are not asbestos must be confirmed by EDXA or measurement of a zone axis diffraction pattern.
- h. Fibers classified as chrysotile must be identified by diffraction or X-ray analysis and recorded on a count sheet. X-ray analysis alone can be used only after 70 s/mm <sup>2</sup> have been exceeded for a particular sample.
- i. Fibers classified as amphiboles must be identified by X-ray analysis and electron diffraction and recorded on the count sheet. (X-ray analysis alone can be used only after 70 s/mm $^2$  have been exceeded for a particular sample.)
- j. If a diffraction pattern was recorded on film, record the micrograph number on the count sheet.
- k. If an electron diffraction was attempted but no pattern was observed, record N on the count sheet.
- I. If an EDXA spectrum was attempted but not observed, record N on the count sheet.
- m. If an X-ray analysis spectrum is stored, record the file and disk number on the count sheet.
- 10. Classification Rules.
- a. Fiber. A structure having a minimum length greater than or equal to  $0.5 \, \mu m$  and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.
- b. *Bundle*. A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.
- c. *Cluster.* A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.
- d. *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.
- 11. After finishing with a grid, remove it from the microscope, and replace it in the appropriate grid holder. Sample grids must be stored for a minimum of 1 year from the date of the analysis; the sample cassette must be retained for a minimum of 30 days by the laboratory or returned at the client's request.

### G. Sample Analytical Sequence

- 1. Under the present sampling requirements a minimum of 13 samples is to be collected for the clearance testing of an abatement site. These include five abatement area samples, five ambient samples, two field blanks, and one sealed blank.
- 2. Carry out visual inspection of work site prior to air monitoring.
- 3. Collect a minimum of 5 air samples inside the work site and 5 samples outside the work site. The indoor and outdoor samples shall be taken during the same time period.
- 4. Remaining steps in the analytical sequence are contained in Unit IV of this Appendix.

## H. Reporting

- 1. The following information must be reported to the client for each sample analyzed:
- a. Concentration in structures per square millimeter and structures per cubic centimeter.
- b. Analytical sensitivity used for the analysis.
- c. Number of asbestos structures.
- d. Area analyzed.
- e. Volume of air sampled (which must be initially supplied to lab by client).
- f. Copy of the count sheet must be included with the report.
- g. Signature of laboratory official to indicate that the laboratory met specifications of the method.
- h. Report form must contain official laboratory identification (e.g., letterhead).
- i. Type of asbestos.

## I. Quality Control/Quality Assurance Procedures (Data Quality Indicators)

Monitoring the environment for airborne asbestos requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards are to be performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way, the quality of the data is defined and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the following Table III:

Unit Operation	OC Check	Emquency	Conformance Expectation
Sample receiving	Review of receiving report	Each sample	95% complete
Sample custody	Review of chain-of-custody record	Each sample	95% complexe
Sample preparation	Supplies and reagents	On receipt	Most specs, or reject.
	Grid opening size	20 openings/20 grids/lot of 1000 or 1 opening/sample	100%
	Special clean area monitoring	After cleaning or service	Most specs or reclean
	Laboratory blank	1 per prop series or 10%	Meet spees, or manufyze series
	Plasma etch blank	1 per 20 samples	75%
	Multiple preps (3 per sample)	Each sample	One with cover of 15 complete grid sqs.
Sample analysis	System check	Each day	Each day
	Alignment check	Each day	Each day
	Magnification calibration with low and high standards	Each month or after service	95%
	ED calibration by gold standard	Weekly	95%
	EDS calibration by copper line	Daily	95%
Performance check	Laboratory blank (measure of cleantiness)	Prop 1 per series or 10% read 1 per 25 samples	Meet specs or reanalyze series
	Replicate counting (measure of precision)	1 per 100 samples	1.5 x Poisson Sul. Dev.
	Duplicate analysis (measure of reproducibility)	1 per 100 samples	2 x Poisson Std. Dev.
	Known samples of typical materials (working standards)	Training and for com- parison with unknowns	100%
	Analysis of NBS SRM 1876 and/or RM 8410 (measure of accuracy and comparability)	1 per analyst per year	1.5 x Poisson Suf. Dev.
	Data entry review (data validation and measure of completeness)	Each sample	95%
	Record and verify ID electron diffraction pattern of structure	1 per 5 samples	80% ассилсу
Calculations and data reduction	Hand calculation of automated data reduction procedure or independent recalculation of hand- calculated data	1 per 100 samples	85%

- 1. When the samples arrive at the laboratory, check the samples and documentation for completeness and requirements before initiating the analysis.
- 2. Check all laboratory reagents and supplies for acceptable asbestos background levels.
- 3. Conduct all sample preparation in a clean room environment monitored by laboratory blanks. Testing with blanks must also be done after cleaning or servicing the room.
- 4. Prepare multiple grids of each sample.
- 5. Provide laboratory blanks with each sample batch. Maintain a cumulative average of these results. If there are more than 53 fibers/mm <sup>2</sup> per 10 200-mesh grid openings, the system must be checked for possible sources of contamination.
- 6. Perform a system check on the transmission electron microscope daily.
- 7. Make periodic performance checks of magnification, electron diffraction and energy dispersive X-ray systems as set forth in Table III under Unit II.I.
- 8. Ensure qualified operator performance by evaluation of replicate analysis and standard sample comparisons as set forth in Table III under Unit II.I.
- 9. Validate all data entries.
- 10. Recalculate a percentage of all computations and automatic data reduction steps as specified in Table III under Unit II.I.
- 11. Record an electron diffraction pattern of one asbestos structure from every five samples that contain asbestos. Verify the identification of the pattern by measurement or comparison of the pattern with patterns collected from standards under the same conditions. The records must also demonstrate that the identification of the pattern has been verified by a qualified individual and that the operator who made the identification is maintaining at least an 80 percent correct visual identification based on his measured patterns.

12. Appropriate logs or records must be maintained by the analytical laboratory verifying that it is in compliance with the mandatory quality assurance procedures.

#### J. References

For additional background information on this method, the following references should be consulted.

- 1. "Guidance for Controlling Asbestos-Containing Materials in Buildings," EPA 560/5-85-024, June 1985.
- 2. "Measuring Airborne Asbestos Following an Abatement Action," USEPA, Office of Pollution Prevention and Toxics, EPA 600/4-85-049, 1985.
- 3. Small, John and E. Steel. Asbestos Standards: Materials and Analytical Methods. N.B.S. Special Publication 619, 1982.
- 4. Campbell, W.J., R.L. Blake, L.L. Brown, E.E. Cather, and J.J. Sjoberg. Selected Silicate Minerals and Their Asbestiform Varieties. Information Circular 8751, U.S. Bureau of Mines, 1977.
- 5. Quality Assurance Handbook for Air Pollution Measurement System. Ambient Air Methods, EPA 600/4-77-027a, USEPA, Office of Research and Development, 1977.
- 6. Method 2A: Direct Measurement of Gas Volume through Pipes and Small Ducts. 40 CFR Part 60 Appendix A.
- 7. Burdette, G.J., Health & Safety Exec. Research & Lab. Services Div., London, "Proposed Analytical Method for Determination of Asbestos in Air."
- 8. Chatfield, E.J., Chatfield Tech. Cons., Ltd., Clark, T., PEI Assoc., "Standard Operating Procedure for Determination of Airborne Asbestos Fibers by Transmission Electron Microscopy Using Polycarbonate Membrane Filters," WERL SOP 87-1, March 5, 1987.
- 9. NIOSH Method 7402 for Asbestos Fibers, 12-11-86 Draft.
- 10. Yamate, G., Agarwall, S.C., Gibbons, R.D., IIT Research Institute, "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy," Draft report, USEPA Contract 68-02-3266, July 1984.
- 11. "Guidance to the Preparation of Quality Assurance Project Plans," USEPA, Office of Pollution Prevention and Toxics, 1984.

## III. Nonmandatory Transmission Electron Microscopy Method

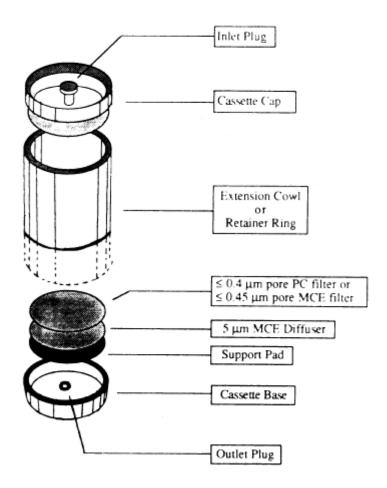
### A. Definitions of Terms

- 1. Analytical sensitivity -- Airborne asbestos concentration represented by each fiber counted under the electron microscope. It is determined by the air volume collected and the proportion of the filter examined. This method requires that the analytical sensitivity be no greater than  $0.005 \text{ s/cm}^3$ .
- 2. *Asbestiform* -- A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.
- 3. Aspect ratio -- A ratio of the length to the width of a particle. Minimum aspect ratio as defined by this method is equal to or greater than 5:1.

- 4. *Bundle* -- A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.
- 5. Clean area -- A controlled environment which is maintained and monitored to assure a low probability of asbestos contamination to materials in that space. Clean areas used in this method have HEPA filtered air under positive pressure and are capable of sustained operation with an open laboratory blank which on subsequent analysis has an average of less than 18 structures/mm <sup>2</sup> in an area of 0.057 mm <sup>2</sup> (nominally 10 200 mesh grid openings) and a maximum of 53 structures/mm <sup>2</sup> for no more than one single preparation for that same area.
- 6. *Cluster* -- A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.
- 7. ED -- Electron diffraction.
- 8. EDXA -- Energy dispersive X-ray analysis.
- 9. Fiber -- A structure greater than or equal to 0.5  $\mu$ m in length with an aspect ratio (length to width) of 5:1 or greater and having substantially parallel sides.
- 10. *Grid* -- An open structure for mounting on the sample to aid in its examination in the TEM. The term is used here to denote a 200-mesh copper lattice approximately 3 mm in diameter.
- 11. *Intersection* -- Nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater.
- 12. *Laboratory sample coordinator* -- That person responsible for the conduct of sample handling and the certification of the testing procedures.
- 13. Filter background level -- The concentration of structures per square millimeter of filter that is considered indistinguishable from the concentration measured on blanks (filters through which no air has been drawn). For this method the filter background level is defined as 70 structures/mm <sup>2</sup>.
- 14. *Matrix* -- Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.
- 15. NSD -- No structure detected.
- 16. Operator -- A person responsible for the TEM instrumental analysis of the sample.
- 17. *PCM* -- Phase contrast microscopy.
- 18. SAED -- Selected area electron diffraction.
- 19. SEM -- Scanning electron microscope.
- 20. STEM -- Scanning transmission electron microscope.
- 21. Structure -- a microscopic bundle, cluster, fiber, or matrix which may contain asbestos.
- 22. S/cm 3 -- Structures per cubic centimeter.
- 23. *S/mm 2* -- Structures per square millimeter.
- 24. *TEM* -- Transmission electron microscope.

### B. Sampling

- 1. Sampling operations must be performed by qualified individuals completely independent of the abatement contractor to avoid possible conflict of interest (See References 1, 2, and 5 of Unit III.L.) Special precautions should be taken to avoid contamination of the sample. For example, materials that have not been prescreened for their asbestos background content should not be used; also, sample handling procedures which do not take cross contamination possibilities into account should not be used.
- 2. Material and supply checks for asbestos contamination should be made on all critical supplies, reagents, and procedures before their use in a monitoring study.
- 3. Quality control and quality assurance steps are needed to identify problem areas and isolate the cause of the contamination (see Reference 5 of Unit III.L.). Control checks shall be permanently recorded to document the quality of the information produced. The sampling firm must have written quality control procedures and documents which verify compliance. Independent audits by a qualified consultant or firm should be performed once a year. All documentation of compliance should be retained indefinitely to provide a guarantee of quality. A summary of Sample Data Quality Objectives is shown in Table II of Unit II.B.
- 4. Sampling materials.
- a. Sample for airborne asbestos following an abatement action using commercially available cassettes.
- b. Use either a cowling or a filter-retaining middle piece. Conductive material may reduce the potential for particulates to adhere to the walls of the cowl.
- c. Cassettes must be verified as "clean" prior to use in the field. If packaged filters are used for loading or preloaded cassettes are purchased from the manufacturer or a distributor, the manufacturer's name and lot number should be entered on all field data sheets provided to the laboratory, and are required to be listed on all reports from the laboratory.
- d. Assemble the cassettes in a clean facility (See definition of clean area under Unit III.A.).
- e. Reloading of used cassettes is not permitted.
- f. Use sample collection filters which are either polycarbonate having a pore size of less than or equal to  $0.4 \mu m$  or mixed cellulose ester having a pore size of less than or equal to  $0.45 \mu m$ .
- g. Place these filters in series with a backup filter with a pore size of  $5.0 \mu m$  (to serve as a diffuser) and a support pad. See the following Figure 1:



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- h. When polycarbonate filters are used, position the highly reflective face such that the incoming particulate is received on this surface.
- i. Seal the cassettes to prevent leakage around the filter edges or between cassette part joints. A mechanical press may be useful to achieve a reproducible leak-free seal. Shrink fit gel-bands may be used for this purpose and are available from filter manufacturers and their authorized distributors.
- j. Use wrinkle-free loaded cassettes in the sampling operation.
- 5. Pump setup.
- a. Calibrate the sampling pump over the range of flow rates and loads anticipated for the monitoring period with this flow measuring device in series. Perform this calibration using guidance from EPA Method 2A each time the unit is sent to the field (See Reference 6 of Unit III.L.).
- b. Configure the sampling system to preclude pump vibrations from being transmitted to the cassette by using a sampling stand separate from the pump station and making connections with flexible tubing.
- c. Maintain continuous smooth flow conditions by damping out any pump action fluctuations if necessary.

- d. Check the sampling system for leaks with the end cap still in place and the pump operating before initiating sample collection. Trace and stop the source of any flow indicated by the flowmeter under these conditions.
- e. Select an appropriate flow rate equal to or greater than 1 L/min or less than 10 L/min for 25 mm cassettes. Larger filters may be operated at proportionally higher flow rates.
- f. Orient the cassette downward at approximately 45 degrees from the horizontal.
- g. Maintain a log of all pertinent sampling information, such as pump identification number, calibration data, sample location, date, sample identification number, flow rates at the beginning, middle, and end, start and stop times, and other useful information or comments. Use of a sampling log form is recommended. See the following Figure 2:

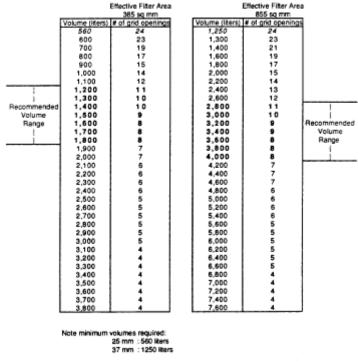
FIGURE 2 -- SAMPLING LOG FORM

Sample Number Location of Sample Pump I.D. Time Middle End Flow Rate

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- h. Initiate a chain of custody procedure at the start of each sampling, if this is requested by the client.
- i. Maintain a close check of all aspects of the sampling operation on a regular basis.
- j. Continue sampling until at least the minimum volume is collected, as specified in the following Table I:

TABLE 1--NUMBER OF 200 MESH EM GRID OPENINGS (0.0057 MM²) THAT NEED TO BE ANALYZED TO MAINTAIN SENSITIVITY OF 0.005 STRUCTURES/CC BASED ON VOLUME AND EFFECTIVE FILTER AREA



Filter diameter of 25 mm = effective area of 385 sq mm Filter diameter of 37 mm = effective area of 855 sq mm

- k. At the conclusion of sampling, turn the cassette upward before stopping the flow to minimize possible particle loss. If the sampling is resumed, restart the flow before reorienting the cassette downward. Note the condition of the filter at the conclusion of sampling.
- I. Double check to see that all information has been recorded on the data collection forms and that the cassette is securely closed and appropriately identified using a waterproof label. Protect cassettes in individual clean resealed polyethylene bags. Bags are to be used for storing cassette caps when they are removed for sampling purposes. Caps and plugs should only be removed or replaced using clean hands or clean disposable plastic gloves.
- m. Do not change containers if portions of these filters are taken for other purposes.
- 6. Minimum sample number per site. A minimum of 13 samples are to be collected for each testing consisting of the following:
- a. A minimum of five samples per abatement area.
- b. A minimum of five samples per ambient area positioned at locations representative of the air entering the abatement site.
- c. Two field blanks are to be taken by removing the cap for not more than 30 sec and replacing it at the time of sampling before sampling is initiated at the following places:
- i. Near the entrance to each ambient area.
- ii. At one of the ambient sites.

(Note: Do not leave the blank open during the sampling period.)

d. A sealed blank is to be carried with each sample set. This representative cassette is not to be opened in the field.

- 7. Abatement area sampling.
- a. Conduct final clearance sampling only after the primary containment barriers have been removed; the abatement area has been thoroughly dried; and, it has passed visual inspection tests by qualified personnel. (See Reference 1 of Unit III.L.)
- b. Containment barriers over windows, doors, and air passageways must remain in place until the TEM clearance sampling and analysis is completed and results meet clearance test criteria. The final plastic barrier remains in place for the sampling period.
- c. Select sampling sites in the abatement area on a random basis to provide unbiased and representative samples.
- d. After the area has passed a thorough visual inspection, use aggressive sampling conditions to dislodge any remaining dust.
- i. Equipment used in aggressive sampling such as a leaf blower and/or fan should be properly cleaned and decontaminated before use.
- ii. Air filtration units shall remain on during the air monitoring period.
- iii. Prior to air monitoring, floors, ceiling and walls shall be swept with the exhaust of a minimum one (1) horsepower leaf blower.
- iv. Stationary fans are placed in locations which will not interfere with air monitoring equipment. Fan air is directed toward the ceiling. One fan shall be used for each 10,000 ft of worksite.
- v. Monitoring of an abatement work area with high-volume pumps and the use of circulating fans will require electrical power. Electrical outlets in the abatement area may be used if available. If no such outlets are available, the equipment must be supplied with electricity by the use of extension cords and strip plug units. All electrical power supply equipment of this type must be approved Underwriter Laboratory equipment that has not been modified. All wiring must be grounded. Ground fault interrupters should be used. Extreme care must be taken to clean up any residual water and ensure that electrical equipment does not become wet while operational.
- vi. Low volume pumps may be carefully wrapped in 6-mil polyethylene to insulate the pump from the air. High volume pumps cannot be sealed in this manner since the heat of the motor may melt the plastic. The pump exhausts should be kept free.
- vii. If recleaning is necessary, removal of this equipment from the work area must be handled with care. It is not possible to completely decontaminate the pump motor and parts since these areas cannot be wetted. To minimize any problems in this area, all equipment such as fans and pumps should be carefully wet wiped prior to removal from the abatement area. Wrapping and sealing low volume pumps in 6-mil polyethylene will provide easier decontamination of this equipment. Use of clean water and disposable wipes should be available for this purpose.
- e. Pump flow rate equal to or greater than 1 L/min or less than 10 L/min may be used for 25 mm cassettes. The larger cassette diameters may have comparably increased flow.
- f. Sample a volume of air sufficient to ensure the minimum quantitation limits. (See Table I of Unit III.B.5.j.)
- 8. Ambient sampling.
- a. Position ambient samplers at locations representative of the air entering the abatement site. If makeup air entering the abatement site is drawn from another area of the building which is outside of the abatement area, place the pumps in the building, pumps should be placed out of

doors located near the building and away from any obstructions that may influence wind patterns. If construction is in progress immediately outside the enclosure, it may be necessary to select another ambient site. Samples should be representative of any air entering the work site.

- b. Locate the ambient samplers at least 3 ft apart and protect them from adverse weather conditions.
- c. Sample same volume of air as samples taken inside the abatement site.

## C. Sample Shipment

- 1. Ship bulk samples in a separate container from air samples. Bulk samples and air samples delivered to the analytical laboratory in the same container shall be rejected.
- 2. Select a rigid shipping container and pack the cassettes upright in a noncontaminating nonfibrous medium such as a bubble pack. The use of resealable polyethylene bags may help to prevent jostling of individual cassettes.
- 3. Avoid using expanded polystyrene because of its static charge potential. Also avoid using particle-based packaging materials because of possible contamination.
- 4. Include a shipping bill and a detailed listing of samples shipped, their descriptions and all identifying numbers or marks, sampling data, shipper's name, and contact information. For each sample set, designate which are the ambient samples, which are the abatement area samples, which are the field blanks, and which is the sealed blank if sequential analysis is to be performed.
- 5. Hand-carry samples to the laboratory in an upright position if possible; otherwise choose that mode of transportation least likely to jar the samples in transit.
- 6. Address the package to the laboratory sample coordinator by name when known and alert him or her of the package description, shipment mode, and anticipated arrival as part of the chain of custody and sample tracking procedures. This will also help the laboratory schedule timely analysis for the samples when they are received.

# D. Quality Control/Quality Assurance Procedures (Data Quality Indicators)

Monitoring the environment for airborne asbestos requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards is performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way, the quality of the data is defined, and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the text below.

- 1. Prescreen the loaded cassette collection filters to assure that they do not contain concentrations of asbestos which may interfere with the analysis of the sample. A filter blank average of less than 18 s/mm $^2$  in an area of 0.057 mm $^2$  (nominally 10 200-mesh grid openings) and a maximum of 53 s/mm $^2$  for that same area for any single preparation is acceptable for this method.
- 2. Calibrate sampling pumps and their flow indicators over the range of their intended use with a recognized standard. Assemble the sampling system with a representative filter -- not the filter which will be used in sampling -- before and after the sampling operation.

- 3. Record all calibration information with the data to be used on a standard sampling form.
- 4. Ensure that the samples are stored in a secure and representative location.
- 5. Ensure that mechanical calibrations from the pump will be minimized to prevent transferral of vibration to the cassette.
- 6. Ensure that a continuous smooth flow of negative pressure is delivered by the pump by installing a damping chamber if necessary.
- 7. Open a loaded cassette momentarily at one of the indoor sampling sites when sampling is initiated. This sample will serve as an indoor field blank.
- 8. Open a loaded cassette momentarily at one of the outdoor sampling sites when sampling is initiated. This sample will serve as an outdoor field blank.
- 9. Carry a sealed blank into the field with each sample series. Do not open this cassette in the field.
- 10. Perform a leak check of the sampling system at each indoor and outdoor sampling site by activating the pump with the closed sampling cassette in line. Any flow indicates a leak which must be eliminated before initiating the sampling operation.
- 11. Ensure that the sampler is turned upright before interrupting the pump flow.
- 12. Check that all samples are clearly labeled and that all pertinent information has been enclosed before transfer of the samples to the laboratory.

# E. Sample Receiving

- 1. Designate one individual as sample coordinator at the laboratory. While that individual will normally be available to receive samples, the coordinator may train and supervise others in receiving procedures for those times when he/she is not available.
- 2. Adhere to the following procedures to ensure both the continued chain-of-custody and the accountability of all samples passing through the laboratory:
- a. Note the condition of the shipping package and data written on it upon receipt.
- b. Retain all bills of lading or shipping slips to document the shipper and delivery time.
- c. Examine the chain-of-custody seal, if any, and the package for its integrity.
- d. If there has been a break in the seal or substantive damage to the package, the sample coordinator shall immediately notify the shipper and a responsible laboratory manager before any action is taken to unpack the shipment.
- e. Packages with significant damage shall be accepted only by the responsible laboratory manager after discussions with the client.
- 3. Unwrap the shipment in a clean, uncluttered facility. The sample coordinator or his or her designee will record the contents, including a description of each item and all identifying numbers or marks. A Sample Receiving Form to document this information is attached for use when necessary. (See the following Figure 3.)

Date of package delivery	Package shipped from						
Carrier	Shipping bill retained						
*Condition of package on receipt							
*Condition of custody seal							
Number of samples received	Shipp	ing mani	fest attache	d			
Purchase Order No.	Projec	aLD					
Comments							
No. Description		npling dian MCE	Sampled Volume Liters	Receiving ID 4	Assigned #		
1							
2							
3							
4		480000					
5							
6							
7	_	_					
8	_						
9	_	_					
10		_					
11		_					
12	_	_					
(Use as many additional sheets as needed.)	411000	_					
Comments							
Date of acceptance into sample bank							
Signature of chain-of-custody recipient							
Disposition of samples							
*Note: If the package has sustained substantial damage manager and the shipper.	e or the o	austody sea	al is broken,	stop and contac	the project		

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**Note:** The person breaking the chain-of-custody seal and itemizing the contents assumes responsibility for the shipment and signs documents accordingly.

- 4. Assign a laboratory number and schedule an analysis sequence.
- 5. Manage all chain-of-custody samples within the laboratory such that their integrity can be ensured and documented.

# F. Sample Preparation

- 1. Personnel not affiliated with the Abatement Contractor shall be used to prepare samples and conduct TEM analysis. Wet-wipe the exterior of the cassettes to minimize contamination possibilities before taking them to the clean sample preparation facility.
- 2. Perform sample preparation in a well-equipped clean facility.

**Note:** The clean area is required to have the following minimum characteristics. The area or hood must be capable of maintaining a positive pressure with make-up air being HEPA filtered. The cumulative analytical blank concentration must average less than  $18 \text{ s/mm}^2$  in an area of  $0.057 \text{ s/mm}^2$  (nominally 10 200-mesh grid openings) with no more than one single preparation to exceed  $53 \text{ s/mm}^2$  for that same area.

3. Preparation areas for air samples must be separated from preparation areas for bulk samples. Personnel must not prepare air samples if they have previously been preparing bulk

samples without performing appropriate personal hygiene procedures, i.e., clothing change, showering, etc.

- 4. *Preparation*. Direct preparation techniques are required. The objective is to produce an intact carbon film containing the particulates from the filter surface which is sufficiently clear for TEM analysis. Currently recommended direct preparation procedures for polycarbonate (PC) and mixed cellulose ester (MCE) filters are described in Unit III.F.7. and 8. Sample preparation is a subject requiring additional research. Variation on those steps which do not substantively change the procedure, which improve filter clearing or which reduce contamination problems in a laboratory are permitted.
- a. Use only TEM grids that have had grid opening areas measured according to directions in Unit III.J.
- b. Remove the inlet and outlet plugs prior to opening the cassette to minimize any pressure differential that may be present.
- c. Examples of techniques used to prepare polycarbonate filters are described in Unit III.F.7.
- d. Examples of techniques used to prepare mixed cellulose ester filters are described in Unit III.F.8.
- e. Prepare multiple grids for each sample.
- f. Store the three grids to be measured in appropriately labeled grid holders or polyethylene capsules.
- 5. Equipment.
- a. Clean area.
- b. Tweezers. Fine-point tweezers for handling of filters and TEM grids.
- c. Scalpel Holder and Curved No. 10 Surgical Blades.
- d. Microscope slides.
- e. Double-coated adhesive tape.
- f. Gummed page reinforcements.
- g. Micro-pipet with disposal tips 10 to 100 µL variable volume.
- h. Vacuum coating unit with facilities for evaporation of carbon. Use of a liquid nitrogen cold trap above the diffusion pump will minimize the possibility of contamination of the filter surface by oil from the pumping system. The vacuum-coating unit can also be used for deposition of a thin film of gold.
- i. *Carbon rod electrodes.* Spectrochemically pure carbon rods are required for use in the vacuum evaporator for carbon coating of filters.
- j. *Carbon rod sharpener*. This is used to sharpen carbon rods to a neck. The use of necked carbon rods (or equivalent) allows the carbon to be applied to the filters with a minimum of heating.
- k. Low-temperature plasma asher. This is used to etch the surface of collapsed mixed cellulose ester (MCE) filters. The asher should be supplied with oxygen, and should be modified as necessary to provide a throttle or bleed valve to control the speed of the vacuum to minimize

disturbance of the filter. Some early models of ashers admit air too rapidly, which may disturb particulates on the surface of the filter during the etching step.

- I. Glass petri dishes, 10 cm in diameter, 1 cm high. For prevention of excessive evaporation of solvent when these are in use, a good seal must be provided between the base and the lid. The seal can be improved by grinding the base and lid together with an abrasive grinding material.
- m. Stainless steel mesh.
- n. Lens tissue.
- o. Copper 200-mesh TEM grids, 3 mm in diameter, or equivalent.
- p. Gold 200-mesh TEM grids, 3 mm in diameter, or equivalent.
- q. Condensation washer.
- r. Carbon-coated, 200-mesh TEM grids, or equivalent.
- s. Analytical balance, 0.1 mg sensitivity.
- t. Filter paper, 9 cm in diameter.
- u. Oven or slide warmer. Must be capable of maintaining a temperature of 65-70 °C.
- v. Polyurethane foam, 6 mm thickness.
- w. Gold wire for evaporation.
- 6. Reagents.
- a. *General*. A supply of ultra-clean, fiber-free water must be available for washing of all components used in the analysis. Water that has been distilled in glass or filtered or deionized water is satisfactory for this purpose. Reagents must be fiber-free.
- b. Polycarbonate preparation method -- chloroform.
- c. Mixed Cellulose Ester (MCE) preparation method -- acetone or the Burdette procedure (Ref. 7 of Unit III.L.).
- 7. TEM specimen preparation from polycarbonate filters.
- a. Specimen preparation laboratory. It is most important to ensure that contamination of TEM specimens by extraneous asbestos fibers is minimized during preparation.
- b. Cleaning of sample cassettes. Upon receipt at the analytical laboratory and before they are taken into the clean facility or laminar flow hood, the sample cassettes must be cleaned of any contamination adhering to the outside surfaces.
- c. Preparation of the carbon evaporator. If the polycarbonate filter has already been carbon-coated prior to receipt, the carbon coating step will be omitted, unless the analyst believes the carbon film is too thin. If there is a need to apply more carbon, the filter will be treated in the same way as an uncoated filter. Carbon coating must be performed with a high-vacuum coating unit. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application, and must not be used. The carbon rods should be sharpened by a carbon rod sharpener to necks of about 4 mm long and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 10 to 12 cm from the surface of a microscope slide held in the rotating and

tilting device.

- d. Selection of filter area for carbon coating. Before preparation of the filters, a 75 mm×50 mm microscope slide is washed and dried. This slide is used to support strips of filter during the carbon evaporation. Two parallel strips of double-sided adhesive tape are applied along the length of the slide. Polycarbonate filters are easily stretched during handling, and cutting of areas for further preparation must be performed with great care. The filter and the MCE backing filter are removed together from the cassette and placed on a cleaned glass microscope slide. The filter can be cut with a curved scalpel blade by rocking the blade from the point placed in contact with the filter. The process can be repeated to cut a strip approximately 3 mm wide across the diameter of the filter. The strip of polycarbonate filter is separated from the corresponding strip of backing filter and carefully placed so that it bridges the gap between the adhesive tape strips on the microscope slide. The filter strip can be held with fine-point tweezers and supported underneath by the scalpel blade during placement on the microscope slide. The analyst can place several such strips on the same microscope slide, taking care to rinse and wet-wipe the scalpel blade and tweezers before handling a new sample. The filter strips should be identified by etching the glass slide or marking the slide using a marker insoluble in water and solvents. After the filter strip has been cut from each filter, the residual parts of the filter must be returned to the cassette and held in position by reassembly of the cassette. The cassette will then be archived for a period of 30 days or returned to the client upon request.
- e. Carbon coating of filter strips. The glass slide holding the filter strips is placed on the rotation-tilting device, and the evaporator chamber is evacuated. The evaporation must be performed in very short bursts, separated by some seconds to allow the electrodes to cool. If evaporation is too rapid, the strips of polycarbonate filter will begin to curl, which will lead to cross-linking of the surface material and make it relatively insoluble in chloroform. An experienced analyst can judge the thickness of carbon film to be applied, and some test should be made first on unused filters. If the film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen. If the coating is too thick, the filter will tend to curl when exposed to chloroform vapor and the carbon film may not adhere to the support mesh. Too thick a carbon film will also lead to a TEM image that is lacking in contrast, and the ability to obtain ED patterns will be compromised. The carbon film should be as thin as possible and remain intact on most of the grid openings of the TEM specimen intact.
- f. Preparation of the Jaffe washer. The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used. A washer consisting of a simple stainless steel bridge is recommended. Several pieces of lens tissue approximately  $1.0~\text{cm} \times 0.5~\text{cm}$  are placed on the stainless steel bridge, and the washer is filled with chloroform to a level where the meniscus contacts the underside of the mesh, which results in saturation of the lens tissue. See References 8 and 10 of Unit III.L.
- g. Placing of specimens into the Jaffe washer. The TEM grids are first placed on a piece of lens tissue so that individual grids can be picked up with tweezers. Using a curved scalpel blade, the analyst excises three 3 mm square pieces of the carbon-coated polycarbonate filter from the filter strip. The three squares are selected from the center of the strip and from two points between the outer periphery of the active surface and the center. The piece of filter is placed on a TEM specimen grid with the shiny side of the TEM grid facing upwards, and the whole assembly is placed boldly onto the saturated lens tissue in the Jaffe washer. If carbon-coated grids are used, the filter should be placed carbon-coated side down. The three excised squares of filters are placed on the same piece of lens tissue. Any number of separate pieces of lens tissue may be placed in the same Jaffe washer. The lid is then placed on the Jaffe washer, and the system is allowed to stand for several hours, preferably overnight.
- h. Condensation washing. It has been found that many polycarbonate filters will not dissolve completely in the Jaffe washer, even after being exposed to chloroform for as long as 3 days. This problem becomes more serious if the surface of the filter was overheated during the carbon evaporation. The presence of undissolved filter medium on the TEM preparation leads to partial or complete obscuration of areas of the sample, and fibers that may be present in these areas of the specimen will be overlooked; this will lead to a low result. Undissolved filter

medium also compromises the ability to obtain ED patterns. Before they are counted, TEM grids must be examined critically to determine whether they are adequately cleared of residual filter medium. It has been found that condensation washing of the grids after the initial Jaffe washer treatment, with chloroform as the solvent, clears all residual filter medium in a period of approximately 1 hour. In practice, the piece of lens tissue supporting the specimen grids is transferred to the cold finger of the condensation washer, and the washer is operated for about 1 hour. If the specimens are cleared satisfactorily by the Jaffe washer alone, the condensation washer step may be unnecessary.

- 8. TEM specimen preparation from MCE filters.
- a. This method of preparing TEM specimens from MCE filters is similar to that specified in NIOSH Method 7402. See References 7, 8, and 9 of Unit III.L.
- b. Upon receipt at the analytical laboratory, the sample cassettes must be cleaned of any contamination adhering to the outside surfaces before entering the clean sample preparation area.
- c. Remove a section from any quadrant of the sample and blank filters.
- d. Place the section on a clean microscope slide. Affix the filter section to the slide with a gummed paged reinforcement or other suitable means. Label the slide with a water and solvent-proof marking pen.
- e. Place the slide in a petri dish which contains several paper filters soaked with 2 to 3 mL acetone. Cover the dish. Wait 2 to 4 minutes for the sample filter to fuse and clear.
- f. Plasma etching of the collapsed filter is required.
- i. The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher. Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the conditions that should be used. This is one area of the method that requires further evaluation. Insufficient etching will result in a failure to expose embedded filters, and too much etching may result in loss of particulate from the surface. As an interim measure, it is recommended that the time for ashing of a known weight of a collapsed filter be established and that the etching rate be calculated in terms of micrometers per second. The actual etching time used for a particular asher and operating conditions will then be set such that a 1-2  $\mu$ m (10 percent) layer of collapsed surface will be removed.
- ii. Place the slide containing the collapsed filters into a low-temperature plasma asher, and etch the filter.
- g. Transfer the slide to a rotating stage inside the bell jar of a vacuum evaporator. Evaporate a  $1~\text{mm}\times5~\text{mm}$  section of graphite rod onto the cleared filter. Remove the slide to a clean, dry, covered petri dish.
- h. Prepare a second petri dish as a Jaffe washer with the wicking substrate prepared from filter or lens paper placed on top of a 6 mm thick disk of clean spongy polyurethane foam. Cut a V-notch on the edge of the foam and filter paper. Use the V-notch as a reservoir for adding solvent. The wicking substrate should be thin enough to fit into the petri dish without touching the lid.
- i. Place carbon-coated TEM grids face up on the filter or lens paper. Label the grids by marking with a pencil on the filter paper or by putting registration marks on the petri dish lid and marking with a waterproof marker on the dish lid. In a fume hood, fill the dish with acetone until the wicking substrate is saturated. The level of acetone should be just high enough to saturate the filter paper without creating puddles.

j. Remove about a quarter section of the carbon-coated filter samples from the glass slides using a surgical knife and tweezers. Carefully place the section of the filter, carbon side down, on the appropriately labeled grid in the acetone-saturated petri dish. When all filter sections have been transferred, slowly add more solvent to the wedge-shaped trough to bring the acetone level up to the highest possible level without disturbing the sample preparations. Cover the petri dish. Elevate one side of the petri dish by placing a slide under it. This allows drops of condensed solvent vapors to form near the edge rather than in the center where they would drip onto the grid preparation.

### G. TEM Method

- 1. Instrumentation.
- a. Use an 80-120 kV TEM capable of performing electron diffraction with a fluorescent screen inscribed with calibrated gradations. If the TEM is equipped with EDXA it must either have a STEM attachment or be capable of producing a spot less than 250 nm in diameter at crossover. The microscope shall be calibrated routinely (see Unit III.J.) for magnification and camera constant.
- b. While not required on every microscope in the laboratory, the laboratory must have either one microscope equipped with energy dispersive X-ray analysis or access to an equivalent system on a TEM in another laboratory. This must be an Energy Dispersive X-ray Detector mounted on TEM column and associated hardware/software to collect, save, and read out spectral information. Calibration of Multi-Channel Analyzer shall be checked regularly for A1 at 1.48 KeV and Cu at 8.04 KeV, as well as the manufacturer's procedures.
- i. Standard replica grating may be used to determine magnification (e.g., 2160 lines/mm).
- ii. Gold standard may be used to determine camera constant.
- c. Use a specimen holder with single tilt and/or double tilt capabilities.
- 2. Procedure.
- a. Start a new Count Sheet for each sample to be analyzed. Record on count sheet: analyst's initials and date; lab sample number; client sample number microscope identification; magnification for analysis; number of predetermined grid openings to be analyzed; and grid identification. See the following Figure 4:

			FIGU	RE 4C0	OUNT SHE	ET			
Lab Sampl	le No.		Filer Type			Open	dor		
Client Sample No.			Filter Area.			Date			
Instrument	I.D.		Orid I.D. Commons						
Magnificat	ion		Grid Openin	g (GO) Area .					
Acc. Volta	úz.		No. GO to b	e Analyzed _					
00	Structure	Structure	Ler	acceptant a			ED Observation		
	No.	Type.	< 5µm	25µm	Chrys.	Argh.	Nonash.	Neg. ID	EDAX
								_	
			L					_	
			L	L		L	L		
			T						
			_						-
								_	
				L					
				<u> </u>			L		
	-	-				ED Obse	ravios.	-	
00	Structure No.	Structure Type*	< Sum	gon L≥5µm	Chrys.	Amph.	Nesash.	Neg. ID	IDAX
	1441	1,750.	5.30m	E 2 pm	C038	Arigin	(Vender)	Neg. 1D	
								-	
					_				
					-			-	
								-	
*B = Bundle C = Cluste			0 = No fibers o						

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- b. Check that the microscope is properly aligned and calibrated according to the manufacturer's specifications and instructions.
- c. Microscope settings: 80-120 kV, grid assessment 250-1000X, then 15,000-20,000X screen magnification for analysis.
- d. Approximately one-half (0.5) of the predetermined sample area to be analyzed shall be performed on one sample grid preparation and the remaining half on a second sample grid preparation.
- e. Determine the suitability of the grid.
- i. Individual grid openings with greater than 5 percent openings (holes) or covered with greater than 25 percent particulate matter or obviously having nonuniform loading shall not be analyzed.
- ii. Examine the grid at low magnification (<1000X) to determine its suitability for detailed study at higher magnifications.
- iii. Reject the grid if:
- (1) Less than 50 percent of the grid openings covered by the replica are intact.
- (2) It is doubled or folded.
- (3) It is too dark because of incomplete dissolution of the filter.

- iv. If the grid is rejected, load the next sample grid.
- v. If the grid is acceptable, continue on to Step 6 if mapping is to be used; otherwise proceed to Step 7.
- f. Grid Map (Optional).
- i. Set the TEM to the low magnification mode.
- ii. Use flat edge or finder grids for mapping.
- iii. Index the grid openings (fields) to be counted by marking the acceptable fields for one-half (0.5) of the area needed for analysis on each of the two grids to be analyzed. These may be marked just before examining each grid opening (field), if desired.
- iv. Draw in any details which will allow the grid to be properly oriented if it is reloaded into the microscope and a particular field is to be reliably identified.
- g. Scan the grid.
- i. Select a field to start the examination.
- ii. Choose the appropriate magnification (15,000 to 20,000X screen magnification).
- iii. Scan the grid as follows.
- (1) At the selected magnification, make a series of parallel traverses across the field. On reaching the end of one traverse, move the image one window and reverse the traverse.

**Note:** A slight overlap should be used so as not to miss any part of the grid opening (field).

- (2) Make parallel traverses until the entire grid opening (field) has been scanned.
- h. Identify each structure for appearance and size.
- i. Appearance and size: Any continuous grouping of particles in which an asbestos fiber within aspect ratio greater than or equal to 5:1 and a length greater than or equal to 0.5  $\mu$ m is detected shall be recorded on the count sheet. These will be designated asbestos structures and will be classified as fibers, bundles, clusters, or matrices. Record as individual fibers any contiguous grouping having 0, 1, or 2 definable intersections. Groupings having more than 2 intersections are to be described as cluster or matrix. See the following Figure 5:

Count as 1 fiber; 1 Structure; no intersections.



Count as 2 fibers if space between fibers is greater than width of 1 fiber diameter or number of intersections is equal to or less than 1.



Count as 3 structures if space between fibers is greater than width of 1 fiber diameter or if the number of intersections is equal to or less than 2.



Count bundles as 1 structure: 3 or more parallel fibrils less than 1 fiber diameter separation.



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Count clusters as 1 structure; fibers having greater than or equal to 3 intersections.



Count matrix as 1 structure.



DO NOT COUNT AS STRUCTUPES:



Fiber protrusion <5:1 Aspect Ratio



No fiber protusion



Fiber protrusion <0.5 micrometer

<0.5 micrometer in length
 <5:1 Aspect Ratio</pre>

An intersection is a non-parallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater. Combinations such as a matrix and cluster, matrix and bundle, or bundle and cluster are categorized by the dominant fiber quality -- cluster, bundle, and matrix, respectively. Separate categories will be maintained for fibers less than 5  $\mu$ m and for fibers greater than or equal to 5  $\mu$ m in length. Not required, but useful, may be to record the fiber length in 1  $\mu$ m intervals. (Identify each structure morphologically and analyze it as it enters the "window".)

- (1) Fiber. A structure having a minimum length greater than 0.5  $\mu$ m and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed, no intersections.
- (2) *Bundle.* A structure composed of 3 or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.
- (3) *Cluster.* A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings must have more than 2 intersections.
- (4) *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.
- (5) NSD. Record NSD when no structures are detected in the field.
- (6) *Intersection.* Non-parallel touching or crossing of fibers, with the projection having an aspect ratio 5:1 or greater.
- ii. Structure Measurement.
- (1) Recognize the structure that is to be sized.
- (2) Memorize its location in the "window" relative to the sides, inscribed square and to other particulates in the field so this exact location can be found again when scanning is resumed.
- (3) Measure the structure using the scale on the screen.
- (4) Record the length category and structure type classification on the count sheet after the field number and fiber number.
- (5) Return the fiber to its original location in the window and scan the rest of the field for other fibers; if the direction of travel is not remembered, return to the right side of the field and begin the traverse again.
- i. Visual identification of Electron Diffraction (ED) patterns is required for each asbestos structure counted which would cause the analysis to exceed the 70 s/mm <sup>2</sup> concentration. (Generally this means the first four fibers identified as asbestos must exhibit an identifiable diffraction pattern for chrysotile or amphibole.)
- i. Center the structure, focus, and obtain an ED pattern. (See Microscope Instruction Manual for more detailed instructions.)
- ii. From a visual examination of the ED pattern, obtained with a short camera length, classify the observed structure as belonging to one of the following classifications: chrysotile, amphibole, or nonasbestos.
- (1) Chrysotile: The chrysotile asbestos pattern has characteristic streaks on the layer lines other than the central line and some streaking also on the central line. There will be spots of

normal sharpness on the central layer line and on alternate lines (2nd, 4th, etc.). The repeat distance between layer lines is 0.53 nm and the center doublet is at 0.73 nm. The pattern should display (002), (110), (130) diffraction maxima; distances and geometry should match a chrysotile pattern and be measured semiquantitatively.

- (2) Amphibole Group [includes grunerite (amosite), crocidolite, anthophyllite, tremolite, and actinolite]: Amphibole asbestos fiber patterns show layer lines formed by very closely spaced dots, and the repeat distance between layer lines is also about 0.53 nm. Streaking in layer lines is occasionally present due to crystal structure defects.
- (3) Nonasbestos: Incomplete or unobtainable ED patterns, a nonasbestos EDXA, or a nonasbestos morphology.
- iii. The micrograph number of the recorded diffraction patterns must be reported to the client and maintained in the laboratory's quality assurance records. The records must also demonstrate that the identification of the pattern has been verified by a qualified individual and that the operator who made the identification is maintaining at least an 80 percent correct visual identification based on his measured patterns. In the event that examination of the pattern by the qualified individual indicates that the pattern had been misidentified visually, the client shall be contacted. If the pattern is a suspected chrysotile, take a photograph of the diffraction pattern at 0 degrees tilt. If the structure is suspected to be amphibole, the sample may have to be tilted to obtain a simple geometric array of spots.
- j. Energy Dispersive X-Ray Analysis (EDXA).
- i. Required of all amphiboles which would cause the analysis results to exceed the 70 s/mm <sup>2</sup> concentration. (Generally speaking, the first 4 amphiboles would require EDXA.)
- ii. Can be used alone to confirm chrysotile after the 70 s/mm <sup>2</sup> concentration has been exceeded.
- iii. Can be used alone to confirm all nonasbestos.
- iv. Compare spectrum profiles with profiles obtained from asbestos standards. The closest match identifies and categorizes the structure.
- v. If the EDXA is used for confirmation, record the properly labeled spectrum on a computer disk, or if a hard copy, file with analysis data.
- vi. If the number of fibers in the nonasbestos class would cause the analysis to exceed the 70 s/mm $^2$  concentration, their identities must be confirmed by EDXA or measurement of a zone axis diffraction pattern to establish that the particles are nonasbestos.
- k. Stopping Rules.
- i. If more than 50 asbestiform structures are counted in a particular grid opening, the analysis may be terminated.
- ii. After having counted 50 asbestiform structures in a minimum of 4 grid openings, the analysis may be terminated. The grid opening in which the 50th fiber was counted must be completed.
- iii. For blank samples, the analysis is always continued until 10 grid openings have been analyzed.
- iv. In all other samples the analysis shall be continued until an analytical sensitivity of 0.005 s/cm  $^3$  is reached.
- I. Recording Rules. The count sheet should contain the following information:

- i. Field (grid opening): List field number.
- ii. Record "NSD" if no structures are detected.
- iii. Structure information.
- (1) If fibers, bundles, clusters, and/or matrices are found, list them in consecutive numerical order, starting over with each field.
- (2) Length. Record length category of asbestos fibers examined. Indicate if less than 5  $\mu m$  or greater than or equal to 5  $\mu m$ .
- (3) Structure Type. Positive identification of asbestos fibers is required by the method. At least one diffraction pattern of each fiber type from every five samples must be recorded and compared with a standard diffraction pattern. For each asbestos fiber reported, both a morphological descriptor and an identification descriptor shall be specified on the count sheet.
- (4) Fibers classified as chrysotile must be identified by diffraction and/or X-ray analysis and recorded on the count sheet. X-ray analysis alone can be used as sole identification only after 70s/mm <sup>2</sup> have been exceeded for a particular sample.
- (5) Fibers classified as amphiboles must be identified by X-ray analysis and electron diffraction and recorded on the count sheet. (X-ray analysis alone can be used as sole identification only after 70s/mm<sup>2</sup> have been exceeded for a particular sample.)
- (6) If a diffraction pattern was recorded on film, the micrograph number must be indicated on the count sheet.
- (7) If an electron diffraction was attempted and an appropriate spectra is not observed, N should be recorded on the count sheet.
- (8) If an X-ray analysis is attempted but not observed, N should be recorded on the count sheet.
- (9) If an X-ray analysis spectrum is stored, the file and disk number must be recorded on the count sheet.
- m. Classification Rules.
- i. Fiber. A structure having a minimum length greater than or equal to 0.5  $\mu$ m and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.
- ii. *Bundle*. A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.
- iii. *Cluster.* A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.
- iv. *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.
- v. NSD. Record NSD when no structures are detected in the field.
- n. After all necessary analyses of a particle structure have been completed, return the goniometer stage to 0 degrees, and return the structure to its original location by recall of the original location.

- o. Continue scanning until all the structures are identified, classified and sized in the field.
- p. Select additional fields (grid openings) at low magnification; scan at a chosen magnification (15,000 to 20,000X screen magnification); and analyze until the stopping rule becomes applicable.
- q. Carefully record all data as they are being collected, and check for accuracy.
- r. After finishing with a grid, remove it from the microscope, and replace it in the appropriate grid hold. Sample grids must be stored for a minimum of 1 year from the date of the analysis; the sample cassette must be retained for a minimum of 30 days by the laboratory or returned at the client's request.

## H. Sample Analytical Sequence

- 1. Carry out visual inspection of work site prior to air monitoring.
- 2. Collect a minimum of five air samples inside the work site and five samples outside the work site. The indoor and outdoor samples shall be taken during the same time period.
- 3. Analyze the abatement area samples according to this protocol. The analysis must meet the  $0.005 \text{ s/cm}^3$  analytical sensitivity.
- 4. Remaining steps in the analytical sequence are contained in Unit IV. of this Appendix.

# I. Reporting

The following information must be reported to the client. See the following Table II:

TABLE II--EXAMPLE LABORATORY LETTERHEAD

Laboratory	Client		FILTER	Arabood	Sample		
I.D. 1.D.	Type	Diameter, met	Effective Areamen 2	Pore Size, µm	Analyzed Ana, mm <sup>2</sup>	Sample Volume, co	
							-
			-				_

#### INDIVIDUAL ANALYTICAL RESULTS

Laboratory	Client	# Asbestos	Analytical	CONCENTRATION			
I,D,	LD.	Structures	Sensitivity, s/cr	Structures/mm <sup>2</sup>	Structures/cc		
				<del></del>			
			_				
		-					
		<del> </del>					
		-		-			
		<del>                                     </del>					

The analysis was carried out to the approved TEM method. This laboratory is in compliance with the quality specified by the method.

Authorized Signature

- 1. Concentration in structures per square millimeter and structures per cubic centimeter.
- 2. Analytical sensitivity used for the analysis.
- 3. Number of asbestos structures.
- 4. Area analyzed.
- 5. Volume of air samples (which was initially provided by client).
- 6. Average grid size opening.
- 7. Number of grids analyzed.
- 8. Copy of the count sheet must be included with the report.
- 9. Signature of laboratory official to indicate that the laboratory met specifications of the AHERA method.
- 10. Report form must contain official laboratory identification (e.g., letterhead).
- 11. Type of asbestos.

## J. Calibration Methodology

**Note:** Appropriate implementation of the method requires a person knowledgeable in electron diffraction and mineral identification by ED and EDXA. Those inexperienced laboratories wishing to develop capabilities may acquire necessary knowledge through analysis of appropriate standards and by following detailed methods as described in References 8 and 10 of Unit III.L.

- 1. Equipment Calibration. In this method, calibration is required for the air-sampling equipment and the transmission electron microscope (TEM).
- a. *TEM Magnification*. The magnification at the fluorescent screen of the TEM must be calibrated at the grid opening magnification (if used) and also at the magnification used for fiber counting. This is performed with a cross grating replica. A logbook must be maintained, and the dates of calibration depend on the past history of the particular microscope; no frequency is specified. After any maintenance of the microscope that involved adjustment of the power supplied to the lenses or the high-voltage system or the mechanical disassembly of the electron optical column apart from filament exchange, the magnification must be recalibrated. Before the TEM calibration is performed, the analyst must ensure that the cross grating replica is placed at the same distance from the objective lens as the specimens are. For instruments that incorporate an eucentric tilting specimen stage, all speciments and the cross grating replica must be placed at the eucentric position.
- b. Determination of the TEM magnification on the fluorescent screen.
- i. Define a field of view on the fluorescent screen either by markings or physical boundaries. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).
- ii. Insert a diffraction grating replica (for example a grating containing 2,160 lines/mm) into the specimen holder and place into the microscope. Orient the replica so that the grating lines fall perpendicular to the scale on the TEM fluorescent screen. Ensure that the goniometer stage tilt is 0 degrees.

iii. Adjust microscope magnification to 10,000X or 20,000X. Measure the distance (mm) between two widely separated lines on the grating replica. Note the number of spaces between the lines. Take care to measure between the same relative positions on the lines (e.g., between left edges of lines).

**Note**: The more spaces included in the measurement, the more accurate the final calculation. On most microscopes, however, the magnification is substantially constant only within the central 8-10 cm diameter region of the fluorescent screen.

iv. Calculate the true magnification (M) on the fluorescent screen:

M=XG/Y

where:

X=total distance (mm) between the designated grating lines;

G=calibration constant of the grating replica (lines/mm):

Y=number of grating replica spaces counted along X.

- c. Calibration of the EDXA System. Initially, the EDXA system must be calibrated by using two reference elements to calibrate the energy scale of the instrument. When this has been completed in accordance with the manufacturer's instructions, calibration in terms of the different types of asbestos can proceed. The EDXA detectors vary in both solid angle of detection and in window thickness. Therefore, at a particular accelerating voltage in use on the TEM, the count rate obtained from specific dimensions of fiber will vary both in absolute X-ray count rate and in the relative X-ray peak heights for different elements. Only a few minerals are relevant for asbestos abatement work, and in this procedure the calibration is specified in terms of a "fingerprint" technique. The EDXA spectra must be recorded from individual fibers of the relevant minerals, and identifications are made on the basis of semiquantitative comparisons with these reference spectra.
- d. Calibration of Grid Openings.
- i. Measure 20 grid openings on each of 20 random 200-mesh copper grids by placing a grid on a glass slide and examining it under the PCM. Use a calibrated graticule to measure the average field diameter and use this number to calculate the field area for an average grid opening. Grids are to be randomly selected from batches up to 1,000.

Note: A grid opening is considered as one field.

- ii. The mean grid opening area must be measured for the type of specimen grids in use. This can be accomplished on the TEM at a properly calibrated low magnification or on an optical microscope at a magnification of approximately 400X by using an eyepiece fitted with a scale that has been calibrated against a stage micrometer. Optical microscopy utilizing manual or automated procedures may be used providing instrument calibration can be verified.
- e. Determination of Camera Constant and ED Pattern Analysis.
- i. The camera length of the TEM in ED operating mode must be calibrated before ED patterns on unknown samples are observed. This can be achieved by using a carbon-coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film.
- ii. In practice, it is desirable to optimize the thickness of the gold film so that only one or two

sharp rings are obtained on the superimposed ED pattern. Thicker gold film would normally give multiple gold rings, but it will tend to mask weaker diffraction spots from the unknown fibrous particulates. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings are unnecessary on zone-axis ED patterns. An average camera constant using multiple gold rings can be determined. The camera constant is one-half the diameter, D, of the rings times the interplanar spacing, d, of the ring being measured.

# K. Quality Control/Quality Assurance Procedures (Data Quality Indicators)

Monitoring the environment for airborne asbestos requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards is performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way, the quality of the data is defined and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the following Table III:

TABLE	SILL	SUMMAR	Y OF	LABORATORY
	DATA	QUALITY	OBJE	CTIVES

_Unit Operation	OC Check	Fraguency	Conformance Expecution
Sample receiving	Review of receiving report	Each sample	95% complete
Sample custody	Review of chain-of-custody record	Each sample	95% complete
Sample preparation	Supplies and reagents	On receipt	Meet spees, or reject
	Grid opening size	20 openings/20 grids/for of 1000 or 1 opening/sample	100%
	Special clean area monitoring	After cleaning or service	Meet spees or reclean
	Laboratory blank	1 per prep series or 10%	Meet speck, or manalyze series
	Plasmo eich blank	1 per 20 samples	75%
	Multiple preps (3 per sample)	Each sample	One with cover of 15 complete grid sqs.
Sample analysis	System check	Each day	Each day
	Alignment chack	Each day	Each day
	Magnification calibration with low and high standards	Each month or after service	95%
	ED calibration by gold standard	Weekly	95%
	EDS calibration by copper line	Daily	95%
Performance check	Laboratory blank (measure of cleanliness)	Prep 1 per series or 10% read 1 per 25 samples	Meet specs or reanalyze series
	Replicate counting (measure of precision)	l per 100 samples	1.5 x Poisson Sul. Dev.
	Duplicate analysis (measure of reproducibility)	1 per 100 samples	2 x Poisson Std. Dev.
	Known samples of typical materials (working standards)	Training and for com- parison with unknowns	100%
	Analysis of NBS SRM 1876 and/or RM 8410 (measure of accuracy and comparability)	I per analyst per year	1.5 x Poisson Std. Dev.
	Data entry review (data validation and measure of completeness)	Each sample	95%
	Record and verify ID electron diffraction pattern of structure	1 per 5 samples	80% accuracy
Calculations and data reduction	Hand calculation of automated data reduction procedure or independent recalculation of hand- calculated data	I per 100 samples	85%

- 1. When the samples arrive at the laboratory, check the samples and documentation for completeness and requirements before initiating the analysis.
- 2. Check all laboratory reagents and supplies for acceptable asbestos background levels.
- 3. Conduct all sample preparation in a clean room environment monitored by laboratory blanks and special testing after cleaning or servicing the room.
- 4. Prepare multiple grids of each sample.
- 5. Provide laboratory blanks with each sample batch. Maintain a cumulative average of these results. If this average is greater than 53 f/mm $^2$  per 10 200-mesh grid openings, check the

system for possible sources of contamination.

- 6. Check for recovery of asbestos from cellulose ester filters submitted to plasma asher.
- 7. Check for asbestos carryover in the plasma asher by including a blank alongside the positive control sample.
- 8. Perform a systems check on the transmission electron microscope daily.
- 9. Make periodic performance checks of magnification, electron diffraction and energy dispersive X-ray systems as set forth in Table III of Unit III.K.
- 10. Ensure qualified operator performance by evaluation of replicate counting, duplicate analysis, and standard sample comparisons as set forth in Table III of Unit III.K.
- 11. Validate all data entries.
- 12. Recalculate a percentage of all computations and automatic data reduction steps as specified in Table III.
- 13. Record an electron diffraction pattern of one asbestos structure from every five samples that contain asbestos. Verify the identification of the pattern by measurement or comparison of the pattern with patterns collected from standards under the same conditions.

The outline of quality control procedures presented above is viewed as the minimum required to assure that quality data is produced for clearance testing of an asbestos abated area. Additional information may be gained by other control tests. Specifics on those control procedures and options available for environmental testing can be obtained by consulting References 6, 7, and 11 of Unit III.L.

# L. References

For additional background information on this method the following references should be consulted.

- 1. "Guidelines for Controlling Asbestos-Containing Materials in Buildings," EPA 560/5-85-024, June 1985.
- 2. "Measuring Airborne Asbestos Following an Abatement Action," USEP/Office of Pollution Prevention and Toxics, EPA 600/4-85-049, 1985.
- 3. Small, John and E. Steel. Asbestos Standards: Materials and Analytical Methods. N.B.S. Special Publication 619, 1982.
- 4. Campbell, W.J., R.L. Blake, L.L. Brown, E.E. Cather, and J.J. Sjoberg. Selected Silicate Minerals and Their Asbestiform Varieties. Information Circular 8751, U.S. Bureau of Mines, 1977.
- 5. Quality Assurance Handbook for Air Pollution Measurement System. Ambient Air Methods, EPA 600/4-77-027a, USEPA, Office of Research and Development, 1977.
- 6. Method 2A: Direct Measurement of Gas Volume Through Pipes and Small Ducts. 40 CFR Part 60 Appendix A.
- 7. Burdette, G.J. Health & Safety Exec., Research & Lab. Services Div., London, "Proposed Analytical Method for Determination of Asbestos in Air."
- 8. Chatfield, E.J., Chatfield Tech. Cons., Ltd., Clark, T., PEI Assoc. "Standard Operating Procedure for Determination of Airborne Asbestos Fibers by Transmission Electron Microscopy

Using Polycarbonate Membrane Filters." WERL SOP 87-1, March 5, 1987.

- 9. NIOSH. Method 7402 for Asbestos Fibers, December 11, 1986 Draft.
- 10. Yamate, G., S.C. Agarwall, R.D. Gibbons, IIT Research Institute, "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy." Draft report, USEPA Contract 68-02-3266, July 1984.
- 11. Guidance to the Preparation of Quality Assurance Project Plans. USEPA, Office of Pollution Prevention and Toxics, 1984.

# IV. Mandatory Interpretation of Transmission Electron Microscopy Results to Determine Completion of Response Actions

#### A. Introduction

A response action is determined to be completed by TEM when the abatement area has been cleaned and the airborne asbestos concentration inside the abatement area is no higher than concentrations at locations outside the abatement area. "Outside" means outside the abatement area, but not necessarily outside the building. EPA reasons that an asbestos removal contractor cannot be expected to clean an abatement area to an airborne asbestos concentration that is lower than the concentration of air entering the abatement area from outdoors or from other parts of the building. After the abatement area has passed a thorough visual inspection, and before the outer containment barrier is removed, a minimum of five air samples inside the abatement area and a minimum of five air samples outside the abatement area must be collected. Hence, the response action is determined to be completed when the average airborne asbestos concentration measured inside the abatement area is not statistically different from the average airborne asbestos concentration measured outside the abatement area.

The inside and outside concentrations are compared by the Z-test, a statistical test that takes into account the variability in the measurement process. A minimum of five samples inside the abatement area and five samples outside the abatement area are required to control the false negative error rate, i.e., the probability of declaring the removal complete when, in fact, the air concentration inside the abatement area is significantly higher than outside the abatement area. Additional quality control is provided by requiring three blanks (filters through which no air has been drawn) to be analyzed to check for unusually high filter contamination that would distort the test results.

When volumes greater than or equal to 1,199 L for a 25 mm filter and 2,799 L for a 37 mm filter have been collected and the average number of asbestos structures on samples inside the abatementareaisnogreater than 70 s/mm <sup>2</sup> of filter, the response action may be considered complete without comparing the inside samples to the outside samples. EPA is permitting this initial screening test to save analysis costs in situations where the airborne asbestos concentration is sufficiently low so that it cannot be distinguished from the filter contamination/background level (fibers deposited on the filter that are unrelated to the air being sampled). The screening test cannot be used when volumes of less than 1,199 L for 25 mm filter or 2,799 L for a 37 mm filter are collected because the ability to distinguish levels significantly different from filter background is reduced at low volumes.

The initial screening test is expressed in structures per square millimeter of filter because filter background levels come from sources other than the air being sampled and cannot be meaningfully expressed as a concentration per cubic centimeter of air. The value of 70 s/mm <sup>2</sup> is based on the experience of the panel of microscopists who consider one structure in 10 grid openings (each grid opening with an area of 0.0057 mm <sup>2</sup>) to be comparable with contamination/background levels of blank filters. The decision is based, in part, on Poisson statistics which indicate that four structures must be counted on a filter before the fiber count is statistically distinguishable from the count for one structure. As more information on the performance of the method is collected, this criterion may be modified. Since different

combinations of the number and size of grid openings are permitted under the TEM protocol, the criterion is expressed in structures per square millimeter of filter to be consistent across all combinations. Four structures per 10 grid openings corresponds to approximately 70 s/mm<sup>2</sup>.

## B. Sample Collection and Analysis

- 1. A minimum of 13 samples is required: five samples collected inside the abatement area, five samples collected outside the abatement area, two field blanks, and one sealed blank.
- 2. Sampling and TEM analysis must be done according to either the mandatory or nonmandatory protocols in Appendix A. At least 0.057 mm <sup>2</sup> of filter must be examined on blank filters.

# C. Interpretation of Results

- 1. The response action shall be considered complete if either:
- a. Each sample collected inside the abatement area consists of at least 1,199 L of air for a 25 mm filter, or 2,799 L of air for a 37 mm filter, and the arithmetic mean of their asbestos structure concentrations per square millimeter of filter is less than or equal to 70 s/mm $^2$ ; or
- b. The three blank samples have an arithmetic mean of the asbestos structure concentration on the blank filtersthatisless than or equal to 70 s/mm  $^2$  and the average airborne asbestos concentration measured inside the abatement area is not statistically higher than the average airborne asbestos concentration measured outside the abatement area as determined by the Z-test. The Z-test is carried out by calculating

$$Z = \frac{\bar{Y}_1 - \bar{Y}_0}{0.8(1/n_1 + 1/n_0)^{1/2}}$$

where YI is the average of the natural logarithms of the inside samples and YO is the average of the natural logarithms of the outside samples, nI is the number of inside samples and nO is the number of outside samples. The response action is considered complete if Z is less than or equal to 1.65.

**Note**: When no fibers are counted, the calculated detection limit for that analysis is inserted for the concentration.

2. If the abatement site does not satisfy either (1) or (2) of this Section C, the site must be recleaned and a new set of samples collected.

# D. Sequence for Analyzing Samples

It is possible to determine completion of the response action without analyzing all samples. Also, at any point in the process, a decision may be made to terminate the analysis of existing samples, reclean the abatement site, and collect a new set of samples. The following sequence is outlined to minimize the number of analyses needed to reach a decision.

- 1. Analyze the inside samples.
- 2. If at least 1,199 L of air for a 25 mm filter or 2,799 L of air for a 37 mm filter is collected for each inside sample and the arithmetic mean concentration of structures per square millimeter of filter is less than or equal to 70 s/mm $^2$ , the response action is complete and no further analysis is needed.
- 3. If less than 1,199 L of air for a 25 mm filter or 2,799 L of air for a 37 mm filter is collected for any of the inside samples, or the arithmetic mean concentration of structures per square millimeteroffilterisgreaterthan 70 s/mm<sup>2</sup>, analyze the three blanks.

- 4. If the arithmetic mean concentration of structures per square millimeter on the blank filters is greater than 70 s/mm <sup>2</sup>, terminate the analysis, identify and correct the source of blank contamination, and collect a new set of samples.
- 5. If the arithmetic mean concentration of structures per square millimeter on the blank filters is less than or equal to 70 s/mm<sup>2</sup>, analyze the outside samples and perform the Z-test.
- 6. If the Z-statistic is less than or equal to 1.65, the response action is complete. If the Z-statistic is greater than 1.65, reclean the abatement site and collect a new set of samples.

[52 FR 41857, Oct. 30, 1987]

Appendix B to Subpart E of Part 763 [Reserved]



**Sample Custody** 

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S. Budney

Date: March 2007

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E-Signed by Michael C. Malloy

Approved:

Signature/Date

QA Review: Jo Nell Mullins

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VERIEY authenticity with ApproveIt

Issued:

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# 1.0 Objective

Because of the evidentiary nature of samples collected during environmental investigations, possession must be traceable from the time the samples are collected until their derived data are introduced as evidence in legal proceedings. To maintain and document sample possession, sample custody procedures are followed. All paperwork associated with the sample custody procedures will be retained in CDM Federal Programs Corporation (CDM) files unless the client requests that it be transferred to them for use in legal proceedings or at the completion of the contract.

**Note**: Sample custody documentation requirements vary with the specific EPA region or client. This SOP is intended to present basic sample custody requirements, along with common options. Specific sample custody requirements shall be presented in the project-specific quality assurance (QA) project plan or project-specific modification or clarification form (see Section U-1).

# 2.0 Background

### 2.1 Definitions

**Sample** - A sample is material to be analyzed that is contained in single or multiple containers representing a unique sample identification number.

Sample Custody - A sample is under custody if:

- 1. It is in your possession
- 2. It is in your view, after being in your possession
- 3. It was in your possession and you locked it up
- 4. It is in a designated secure area

**Chain-of-Custody Record** - A chain-of-custody record is a form used to document the transfer of custody of samples from one individual to another.

**Custody Seal** - A custody seal is a tape-like seal that is part of the chain-of-custody process and is used to detect tampering with samples after they have been packed for shipping.

**Sample Label** - A sample label is an adhesive label placed on sample containers to designate a sample identification number and other sampling information.

**Sample Tag** - A sample tag is attached with string to a sample container to designate a sample identification number and other sampling information. Tags may be used when it is difficult to physically place adhesive labels on the container (e.g., in the case of small air sampling tubes).

# 3.0 General Responsibilities

**Sampler** - The sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched.

**Field Team Leader** - The field team leader (FTL) is responsible for ensuring that strict chain-of-custody procedures are maintained during all sampling events. The FTL is also responsible for coordinating with the subcontractor laboratory to

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ensure that adequate information is recorded on custody records. The FTL determines whether proper custody procedures were followed during the fieldwork.

**Field Sample Custodian** - The field sample custodian, when designated by the FTL, is responsible for accepting custody of samples from the sampler(s) and properly packing and shipping the samples to the laboratory assigned to do the analyses. A field sample custodian is typically designated only for large and complex field efforts.

**Note**: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/quality assurance project plan (QAPP).

## 4.0 Required Supplies

- Chain-of-custody records (applicable client or CDM forms)
- Sample labels and/or tags
- EPA Field Operations Records Management System II Lite<sup>TM</sup> (FORMS II Lite<sup>TM</sup>) software (if required)
- Printer paper

- Custody seals
- Clear tape
- Computer
- Printer

## 5.0 Procedures

## 5.1 Chain-of-Custody Record

This procedure establishes a method for maintaining custody of samples through use of a chain-of-custody record. This procedure will be followed for all samples collected or split samples accepted.

#### **Field Custody**

- 1. Collect only the number of samples needed to represent the media being sampled. To the extent possible, determine the quantity and types of samples and sample locations before the actual fieldwork. As few people as possible shall handle samples.
- 2. Complete sample labels or tags for each sample using waterproof ink.
- 3. Maintain personal custody of the samples (in your possession) at all times until custody is transferred for sample shipment or directly to the analytical laboratory.

#### **Transfer of Custody and Shipment**

- 1. Complete a chain-of-custody record for all samples (see Figure 1 for an example of a chain-of-custody record. Similar forms may be used when requested by the client). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the sample custodian in the appropriate laboratory.
  - The date/time will be the same for both signatures when custody is transferred directly to another person. When samples are shipped via common carrier (e.g., Federal Express), the date/time will not be the same for both signatures. Common carriers are not required to sign the chain-of-custody record.
  - In all cases, it must be readily apparent that the person who received custody is the same person who relinquished custody to the next custodian.
  - If samples are left unattended or a person refuses to sign, this must be documented and explained on the chainof-custody record.

**Note**: If a field sample custodian has been designated, he/she may initiate the chain-of-custody record, sign, and date as the relinquisher. The individual sampler(s) must sign in the appropriate block, but does (do) not need to sign and date as a relinquisher (refer to Figure 1).

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2. Package samples properly for shipment and dispatch to the appropriate laboratory for analysis. Each shipment must be accompanied by a separate chain-of-custody record. If a shipment consists of multiple coolers, a chain-of-custody record shall be filled out for each cooler documenting only samples contained in that particular cooler.

3. The original record will accompany the shipment, and the copies will be retained by the FTL and, if applicable, distributed to the appropriate sample coordinators. Freight bills will also be retained by the FTL as part of the permanent documentation. The shipping number from the freight bill shall be recorded on the applicable chain-of-custody record and field logbook in accordance with TSOP 4-1, Field Logbook Content and Control.

#### **Procedure for Completing CDM Example Chain-of-Custody Record**

The following procedure is to be used to fill out the CDM chain-of-custody record. The record provided herein (Figure 1) is an example chain-of-custody record. If another type of custody record (i.e., provided by the EPA Contract Laboratory Program (CLP) or a subcontract laboratory or generated by FORMS II Lite™) is used to track the custody of samples, the custody record shall be filled out in its entirety.

- Record project number.
- 2. Record FTL for the project (if a field sample custodian has been designated, also record this name in the "Remarks" box).
- 3. Record the name and address of the laboratory to which samples are being shipped.
- 4. Enter the project name/location or code number.
- 5. Record overnight courier's airbill number.
- 6. Record sample location number.
- 7. Record sample number.
- 8. Note preservatives added to the sample.
- 9. Note media type (matrix) of the sample.
- 10. Note sample type (grab or composite).
- 11. Enter date of sample collection.
- 12. Enter time of sample collection in military time.
- 13. When required by the client, enter the names or initials of the samplers next to the sample location number of the sample they collected.
- 14. List parameters for analysis and the number of containers submitted for each analysis.
- 15. Enter appropriate designation for laboratory quality control (e.g., matrix spike/matrix spike duplicate [MS/MSD], matrix spike/duplicate [MS/D]), or other remarks (e.g., sample depth).
- 16. Sign the chain-of-custody record(s) in the space provided. All samplers must sign each record.
- 17. If sample tags are used, record the sample tag number in the "Remarks" column.
- 18. The originator checks information entered in Items 1 through 16 and then signs the top left "Relinquished by" box, prints his/her name, and enters the current date and time (military).
- 19. Send the top two copies (usually white and yellow) with the samples to the laboratory; retain the third copy (usually pink) for the project files. Retain additional copies for the project file or distribute as required to the appropriate sample coordinators.
- 20. The laboratory sample custodian receiving the sample shipment checks the sample label information against the chain-of-custody record. Sample condition is checked and anything unusual is noted under "Remarks" on the chain-of-custody record. The laboratory custodian receiving custody signs in the adjacent "Received by" box and keeps the copy. The white copy is returned to CDM.

#### 5.2 Sample Labels and Tags

Unless the client directs otherwise, sample labels or tags will be used for all samples collected or accepted for CDM projects.

- 1. Complete one label or tag with the information required by the client for each sample container collected. A typical label or tag would be completed as follows (see Figure 2 for example of sample tag; labels are completed with the equivalent information):
  - Record the project code (i.e., project or task number).
  - Enter the station number (sample number or EPA CLP identification number) if applicable.
  - Record the date to indicate the month, day, and year of sample collection.
  - Enter the time (military) of sample collection.

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- Place a check to indicate composite or grab sample.
- Record the station (sample) location.
- Sign in the space provided.
- Place a check next to "yes" or "no" to indicate if a preservative was added.
- Place a check under "Analyses" next to the parameters for which the sample is to be analyzed. If the desired analysis is not listed, write it in the empty slot. Note: Do not write in the box for "laboratory sample number."
- Place or write additional relevant information under "Remarks."
- 2. Place adhesive labels directly on the sample containers. Place clear tape over the label to protect from moisture.
- 3. Securely attach sample tags to the sample bottle. On 2.27 liter (80 oz.) amber bottles, the tag string may be looped through the ring-style handle and tied. On all other containers, it is recommended that the string be looped around the neck of the bottle, then twisted, and relooped around the neck until the slack in the string is removed.
- 4. Double-check that the information recorded on the sample tag is consistent with the information recorded on the chain-of-custody record.

### 5.3 Custody Seals

Two custody seals must be placed on opposite corners of all shipping containers (e.g., cooler) before shipment. The seals shall be signed and dated by the shipper.

Custody seals may also be required to be placed on individual sample bottles. Check with the client or refer to EPA regional guidelines for direction.

## 5.4 Sample Shipping

CDM Federal SOP 2-1, *Packaging and Shipping Environmental Samples* defines the requirements for packaging and shipping environmental samples.

#### 6.0 Restrictions/Limitations

Check with the EPA region or client for specific guidelines. If no specific guidelines are identified, this procedure shall be followed.

For EPA CLP sampling events, combined chain-of-custody/traffic report forms generated with EPA FORMS II Lite™ or other EPA-specific records may be used. Refer to regional guidelines for completing these forms.

The EPA FORMS II Lite™ software may be used to customize sample labels and custody records when directed by the client or the CDM project manager.

## 7.0 References

U. S. Army Corps of Engineers. 2001. *Requirements for the Preparation of Sampling and Analysis Plan*, EM 200-1-3. Appendix F. February.

U. S. Environmental Protection Agency. Revised March 1992. *National Enforcement Investigations Center, Multi-Media Investigation Manual*, EPA-330/9-89-003-R. p.85.

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	2002. FORMS II Lite™ User's Guide, Version 5.1.
December.	2002. EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, EPA/240/R-02/009. Section 2.2.3
	. 2004. Contract Laboratory Program (CLP). Guidance for Field Samplers. EPA-540-R-00-003. Final. Sectio

3.2. August.

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## Figure 1 **Example CDM Chain-of-Custody Record**

125 Maiden Lane, 5th Floor CHAIN OF CUSTODY CDM New York, NY 10038 (212) 785-9123 Fax: (212) 785-6114 RECORD

PROJECT ID.	F	IELD T	EAM LEAD	DER			BORATORY D ADDRES						DATE	SHIPE	PED
PROJECT NAME/LOC	CATION					1^^	D ADDRES	3					AIRBILL NO.		
						LA	B CONTRA	CT:							
MEDIA TYPE	F	RESER	VATIVES				TYPE								
<ol> <li>Surface Water</li> </ol>		. HCI, p				Grat									
<ol><li>Groundwater</li></ol>			, pH <2		C =	Com	posite	ह स							
3. Leachate			l, pH >12					9 ₽							
Field QC     Soil/Sediment			4, pH <2 cetate, ph					t E							
6. Oil		Line Or		1>9				글용							
7. Waste			reserved					8 8							
8. Other		. Other						ANALYSES (List no. of containers submitted)							
								N 8							
SAMPLE			PRESER-	MEDIA	SAMPLE	20_	TIME	_						BI	EMARKS
LOCATION NO.		MPLE MBER	ADDED	TYPE	TYPE	DATI	SAMPLED								if MS/MSI
1.	11011	- CET	AUULU			-			$\rightarrow$		-		+	_	
2.									-		-	_	+		
3.											$\neg$	+	+		
4.															
5.											$\neg$				
6.															
7.															
8.															
9.															
10.															
SAMPLER SIGNATURES:															
RELINQUISHED BY: (PRINT)	DATE/TIME	PECE	IVED BY:		DATE/I		RELINQUISHE (PRINT)	IDBY:		DATE/TIME	RECE	IVED BY			DATE/TIME
(SIGN)		(SIGN)					(SIGN)				(SIGN)				
RELINQUISHED BY: (PRINT)	DATE/TIME	RECE	IVED BY:		DATE/I		RELINQUISHE (PRINT)	OBY:		DATE/TIME	RECE (PRINT)	IVED BY	1		DATE/TIME
(SIGN)		(8908)					(810%)				(81014)				
COMMENTS:	-														

DISTRIBUTION: White and yellow copies accompany sample shipment to laboratory; yellow copy retained by laboratory. Pink copy retained by samplers.

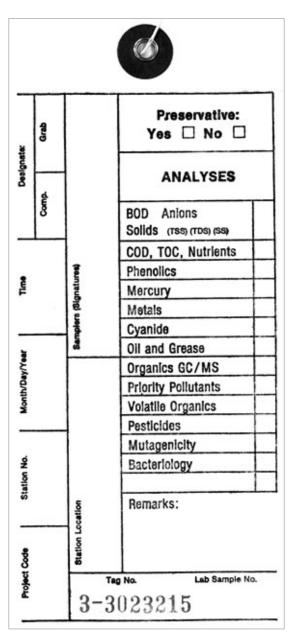
1/98

Note: If requested by the client, different chain-of-custody records may be used. Copies of the template for this record may be obtained from the Chantilly Graphics Department.

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Figure 2 **Example Sample Tag** 



Note: Equivalent sample labels or tags may be used.

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Signature/Date

Prepared: Krista Lippoldt **Technical Review:** Chuck Myers

E-Signed by Michael C. Malloy QA Review: Jo Nell Mullins Approved:

Issued: Signature/Date

## 1.0 Objective

The objective of this SOP is to outline the requirements for the packaging and shipment of environmental samples. Additionally, Sections 2.0 through 7.0 outline requirements for the packaging and shipping of regulated environmental samples under the Department of Transportation (DOT) Hazardous Materials Regulations, the International Air Transportation Association (IATA), and International Civil Aviation Organization (ICAO) Dangerous Goods Regulations for shipment by air and applies only to domestic shipments. This SOP does not cover the requirements for packaging and shipment of equipment (including data loggers and self-contained breathing apparatus [SCBAs] or bulk chemicals that are regulated under the DOT, IATA, and ICAO.

## 1.1 Packaging and Shipping of All Samples

This standard operating procedure (SOP) applies to the packaging and shipping of all environmental samples. If the sample is preserved or radioactive, the following sections may also be applicable.

Section 2.0 - Packaging and Shipping Samples Preserved with Methanol

Section 3.0 - Packaging and Shipping Samples Preserved with Sodium Hydroxide

Section 4.0 - Packaging and Shipping Samples Preserved with Hydrochloric Acid

Section 5.0 - Packaging and Shipping Samples Preserved with Nitric Acid

Section 6.0 - Packaging and Shipping Samples Preserved with Sulfuric Acid

Section 7.0 - Packaging and Shipping Limited-Quantity Radioactive Samples

## 1.2 Background

#### 1.2.1 Definitions

Environmental Sample - An aliquot of air, water, plant material, sediment, or soil that represents the contaminant levels on a site. Samples of potential contaminant sources, like tanks, lagoons, or non-aqueous phase liquids are normally not "environmental" for this purpose. This procedure applies only to environmental samples that contain less than reportable quantities for any foreseeable hazardous constituents according to DOT regulations promulgated in 49 CFR - Part 172.101 Appendix A.

Custody Seal - A custody seal is a narrow adhesive-backed seal that is applied to individual sample containers and/or the container (i.e., cooler) before offsite shipment. Custody seals are used to demonstrate that sample integrity has not been compromised during transportation from the field to the analytical laboratory.

Inside Container - The container, normally made of glass or plastic, that actually contacts the shipped material. Its purpose is to keep the sample from mixing with the ambient environment.

Outside Container - The container, normally made of metal or plastic, that the transporter contacts. Its purpose is to protect the inside container.

Secondary Containment - The outside container provides secondary containment if the inside container breaks (i.e., plastic overpackaging if liquid sample is collected in glass).

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**Excepted Quantity** - Excepted quantities are limits to the mass or volume of a hazardous material in the inside and outside containers below which DOT, IATA, ICAO regulations do not apply. The excepted quantity limits are very low. Most regulated shipments will be made under limited quantity.

**Limited Quantity** - Limited quantity is the maximum amount of a hazardous material below which there are specific labeling or packaging exceptions.

**Performance Testing** - Performance testing is the required testing of outer packaging. These tests include drop and stacking tests.

**Qualified Shipper** - A qualified shipper is a person who has been adequately trained to perform the functions of shipping hazardous materials.

#### 1.2.2 Associated Procedures

■ CDM Federal SOP 1-2, Sample Custody

#### 1.2.3 Discussion

Proper packaging and shipping is necessary to ensure the protection of the integrity of environmental samples shipped for analysis. These shipments are potentially subject to regulations published by DOT, IATA, or ICAO. Failure to abide by these rules places both CDM and the individual employee at risk of serious fines. The analytical holding times for the samples must not be exceeded. The samples shall be packed in time to be shipped for overnight delivery. Make arrangements with the laboratory before sending samples for weekend delivery.

## 1.3 Required Equipment

- Coolers with return address of the appropriate CDM office
- Heavy-duty plastic garbage bags
- Plastic zip-type bags, small and large
- Clear tape
- Nylon reinforced strapping tape
- Duct tape
- Vermiculite (or an equivalent nonflammable material that is inert and absorbent)\*
- Bubble wrap (optional)
- Ice
- Custody seals
- Completed chain-of-custody record or contract laboratory program (CLP) custody records, if applicable
- Completed bill of lading
- "This End Up" and directional arrow labels

\*Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

#### 1.4 Packaging Environmental Samples

The following steps must be followed when packing sample bottles and jars for shipment:

- 1. Verify the samples undergoing shipment meet the definition of "environmental sample" and are not a hazardous material as defined by DOT. Professional judgment and/or consultation with qualified persons such as the appropriate health and safety coordinator or the health and safety manager shall be observed.
- 2. Select a sturdy cooler in good repair. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler. Line the cooler with a large heavy-duty plastic garbage bag.
- 3. Be sure the caps on all bottles are tight (will not leak); check to see that labels and chain-of-custody records are completed properly (SOP 1-2, Sample Custody).
- 4. Place all bottles in separate and appropriately sized plastic zip-top bags and close the bags. Up to three VOA vials may be packed in one bag. Binding the vials together with a rubber band on the outside of the bag, or separating them so that they do not contact each other, will reduce the risk of breakage. Bottles may be wrapped in bubble wrap. Optionally, place three to six VOA vials in a quart metal can and then fill the can with vermiculite or equivalent. *Note*: Trip blanks must be included in coolers containing VOA samples.

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- 5. Place 2 to 4 inches of vermiculite (or equivalent) into a cooler that has been lined with a garbage bag, and then place the bottles and cans in the bag with sufficient space to allow for the addition of packing material between the bottles and cans. It is preferable to place glass sample bottles and jars into the cooler vertically. Glass containers are less likely to break when packed vertically rather than horizontally.
- 6. While placing sample containers into the cooler, conduct an inventory of the contents of the shipping cooler against the chain-of-custody record. The chain-of-custody with the cooler shall reflect only those samples within the cooler.
- 7. Put ice in large plastic zip-top bags (double bagging the zip-tops is preferred) and properly seal. Place the ice bags on top of and/or between the samples. Several bags of ice are required (dependant on outdoor temperature, staging time, etc.) to maintain the cooler temperature at approximately 4° Celsius (C) if the analytical method requires cooling. Fill all remaining space between the bottles or cans with packing material. Securely fasten the top of the large garbage bag with fiber or duct tape.
- 8. Place the completed chain-of-custody record or the CLP traffic report form (if applicable) for the laboratory into a plastic zip-top bag, seal the bag, tape the bag to the inner side of the cooler lid and close the cooler.
- 9. The cooler lid shall be secured with nylon reinforced strapping tape by wrapping each end of the cooler a minimum of two times. Attach a completed chain-of-custody seal across the opening of the cooler on opposite sides. The custody seals shall be affixed to the cooler with half of the seal on the strapping tape so that the cooler cannot be opened without breaking the seal. Complete two more wraps around with fiber tape and place clear tape over the custody seals.
- 10. The shipping container lid must be marked "THIS END UP" and arrow labels that indicate the proper upward position of the container shall be affixed to the cooler. A label containing the name and address of the shipper (CDM) shall be placed on the outside of the container. Labels used in the shipment of hazardous materials (such as Cargo Only Air Craft, Flammable Solids, etc.) are not permitted on the outside of containers used to transport environmental samples and shall not be used. The name and address of the laboratory shall be placed on the container, or when shipping by common courier, the bill of lading shall be completed and attached to the lid of the shipping container.

## 2.0 Packaging and Shipping Samples Preserved with Methanol

#### 2.1 Containers

- The maximum volume of methanol in a sample container is limited to 30 ml.
- The sample container must not be full of methanol.

#### 2.2 Responsibility

It is the responsibility of the qualified shipper to:

- Ensure that the samples undergoing shipment contain no other contaminant that meets the definition of "hazardous material" as defined by DOT
- Determine the amount of preservative in each sample so that accurate determination of quantities can be made

**Note**: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

## 2.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Inner packing may consist of glass or plastic jars
- Outer packaging (for limited quantities) insulated cooler that has passed the ICAO drop test
- Survey documentation (if shipping from Department of Energy [DOE] or radiological sites)
- Class 3 flammable liquid labels
- Orientation labels
- Consignor/consignee labels



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### 2.4 Packaging Samples Preserved with Methanol

The following steps are to be followed when packaging limited-quantity sample shipments:

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape before sampling.
- At a minimum the label must contain:
  - Proiect name - Sample identification number
  - Project number Collector's initials
  - Date and time of sample collection Preservative (note amount of preservative used in miscellaneous section of Sample location the chain-of-custody form)
- Wrap each container (40-ml VOA vials) in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble-wrapped container into a 2.7-mil zip-type bag, removing trapped air.
- Place wrapped containers inside a polyethylene bottle filled with vermiculite; seal the bottle. (Maximum of 4 VOA vials will fit inside a 500-ml wide-mouth polyethylene bottle.)
- Total volume of methanol per shipping container must not exceed 500 ml.
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tieing or taping.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

**Methanol Mixture** UN1230 LTD. QTY.

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Flammable Liquid label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during
- An example of cooler labeling/marking locations is shown in Figure 1.

**Note:** No marking or labeling can be obscured by strapping or duct tape.

Note: The inner packaging of dangerous goods must be placed into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

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Figure 1
Example of Cooler Label/Marking Locations

Strapping To:
Tape

Methanol Mixture
UN1230
LTD. QTY.

Proper Shipping Name and UN Number

Hazard Class Label

## 3.0 Packaging and Shipping Samples Preserved with Sodium Hydroxide 3.1 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

**Excepted Quantities of Sodium Hydroxide Preservatives** 

Preserv	ative		d in Final mple	Quantity of Preservative (ml) for Specified Container					
		рН	Conc.	40 ml	125 ml	250 ml	500 ml	1 L	
NaOH	30%	>12	0.08%		.25	0.5	1	2	

5 drops = 1 ml

## 3.2 Responsibility

It is the responsibility of the qualified shipper to determine the amount of preservative in each sample so that accurate determination of quantities can be made.

**Note**: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

#### 3.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Outer packaging (for limited quantities) insulated cooler that has passed the ICAO drop test
- Inner packings may consist of glass or plastic jars no larger than 1 pint
- Survey documentation (if shipping from DOE or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

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## 3.4 Packaging Samples Preserved with Sodium Hydroxide

Samples containing NaOH as a preservative that exceed the excepted concentration of 0.08 percent (2 ml of a 30 percent NaOH solution per liter) may be shipped as a limited quantity per packing instruction Y819 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are to be followed when packaging limited-quantity samples shipments:

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape before sampling.
- At a minimum the label must contain:
  - Project name Sample identification number
  - Project number Collector's initials
- Date and time of sample collection
   Sample location
   Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form)
- This step is optional; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble-wrapped container into a 2.7-mil zip-type bag, removing trapped air.
- Place glass containers inside a polyethylene bottle filled with vermiculite; seal the bottle.
- The total volume of sample in each cooler must not exceed 1 liter.
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tieing or taping.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

Sodium Hydroxide Solution UN1824 LTD. QTY.

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Corrosive label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marking locations is shown in Figure 1.

**Note**: Samples meeting the exception concentration of 0.08 percent NaOH by weight may be shipped as nonregulated or nonhazardous following the procedure in Section 1.4.

**Note**: No marking or labeling can be obscured by strapping or duct tape.

**Note**: The inner packaging of dangerous goods must be placed into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

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- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

## 4.0 Packaging and Shipping Samples Preserved with Hydrochloric Acid 4.1 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

**Excepted Quantities of Hydrochloric Acid Preservatives** 

Preserv	ative		d in Final mple	Quantity of Preservative (ml) for Specified Container			
		рН	Conc.	40 ml	125 ml	250 ml	
HCI	2N	<1.96	0.04%	.2	.5	1	

5 drops = 1 ml

## 4.2 Responsibility

It is the responsibility of the qualified shipper to:

- Determine the samples undergoing shipment contain no other contaminant that meets the definition of hazardous material as defined by DOT
- Determine the amount of preservative in each sample so that accurate determination of quantities can be made

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

## 4.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3.

- Inner packing may consist of glass or plastic jars no larger than 1 pint.
- Outer packaging (for limited quantities) insulated cooler that has passed the ICAO drop test.
- Survey documentation (if shipping from DOE or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

#### 4.4 Packaging Samples Preserved with Hydrochloric Acid

The following steps are to be followed when packaging limited-quantity sample shipments:

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape before sampling.
- At a minimum the label must contain:
  - Project name Sample identification number
    - Project number Collector's initials
  - Date and time of sample collection Preservative (note amount of preservative used in miscellaneous section of Sample location the chain-of-custody form)
- Wrap each container (40-ml VOA vials) in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble-wrapped container into a 2.7-mil zip-type bag, removing trapped air.
- Place wrapped containers inside a polyethylene bottle filled with vermiculite; seal the bottle. (No more than 4 VOA vials will fit inside a 500-ml wide-mouth polyethylene bottle.)

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- Total volume of sample inside each cooler must not exceed 1 liter.
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tieing or taping.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

Hydrochloric Acid Solution UN1789 LTD. QTY.

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Corrosive label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marking locations is shown in Figure 1.

**Note**: Samples containing less than the exception concentration of 0.04 percent HCl by weight will be shipped as nonregulated or nonhazardous following the procedure in Section 1.4.

Note: No marking or labeling can be obscured by strapping or duct tape.

**Note**: The inner packaging of dangerous goods must be placed into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

# 5.0 Packaging and Shipping Samples Preserved with Nitric Acid5.1 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

#### **Excepted Quantities of Nitric Acid Preservatives**

Preserv	ative		l in Final mple	Quantity of Preservative (ml) for Specified Container						
		pН	Conc.	40 ml	125 ml	250 ml	500 ml	1 L		
HNO <sub>3</sub>	6N	<1.62	0.15%		2	4	5	8		

5 drops = 1 mg/L



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### 5.2 Responsibility

It is the responsibility of the qualified shipper to:

- Determine the samples undergoing shipment contain no other contaminant that meets the definition of hazardous material as defined by DOT
- Determine the amount of preservative in each sample so that accurate determination of quantities can be made

**Note**: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

## 5.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Inner packings may consist of glass or plastic jars no larger than 100 ml.
- Outer packaging (for limited quantities) insulated cooler that has passed the ICAO drop test.
- Survey documentation (if shipping from DOE or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

## 5.4 Packaging Samples Preserved with Nitric Acid

Samples containing HNO<sub>3</sub> as a preservative that exceed the excepted concentration of 0.15 percent HNO<sub>3</sub> will be shipped as a limited quantity per packing instruction Y807 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are to be followed when packaging limited-quantity sample shipments:

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape before sampling.
- At a minimum the label must contain:
  - Project name Sample identification number
  - Project number Collector's initials
  - Date and time of sample collection Preservative (note amount of preservative used in miscellaneous section of
    - Sample location the chain-of-custody form)
- This step is optional; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble-wrapped container into a 2.7-mil zip-type bag, removing trapped air.
- Place glass containers inside a polyethylene bottle filled with vermiculite; seal the bottle.
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tieing or taping.
- The maximum volume of preserved solution in the cooler must not exceed 500 ml.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

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Nitric Acid Solution (with less than 20 percent) UN2031 Ltd. Qty.

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Corrosive label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marking locations is shown in Figure 1.

**Note**: Samples meeting the exception concentration of 0.15 percent HNO<sub>3</sub> by weight will be shipped as nonregulated or nonhazardous following the procedure in Section 1.4.

**Note**: No marking or labeling can be obscured by strapping or duct tape.

**Note**: The inner packaging of dangerous goods must be placed into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

# 6.0 Packaging and Shipping Samples Preserved with Sulfuric Acid 6.1 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

**Excepted Quantities of Sulfuric Acid Preservatives** 

Preserv	/ative		l in Final mple	Quantity of Preservative (ml) for Specified Container					
		pН	Conc.	40 ml	125 ml	250 ml	500 ml	1 L	
H <sub>2</sub> SO <sub>4</sub>	37N	<1.15	0.35%	.1	.25	0.5	1	2	

5 drops = 1 ml

#### 6.2 Responsibility

It is the responsibility of the qualified shipper to:

- Determine the samples undergoing shipment contain no other contaminant that meets the definition of hazardous material as defined by DOT
- Determine the amount of preservative in each sample so that accurate determination of quantities can be made

**Note**: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

#### 6.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

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- Inner packings may consist of glass or plastic jars no larger than 100 ml.
- Outer packaging (for limited quantities) insulated cooler that has passed the ICAO drop test.
- Survey documentation (if shipping from DOE or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

## 6.4 Packaging of Samples Preserved with Sulfuric Acid

Samples containing H<sub>2</sub>SO<sub>4</sub> as a preservative that exceed the excepted concentration of 0.35 percent will be shipped as a limited quantity per packing instruction Y809 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are to be followed when packaging limited-quantity samples shipments:

- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape before sampling.
- At a minimum the label must contain:
  - Project name
- Sample identification number

Project number

- Collector's initials
- Date and time of sample collection
- Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form)

- Sample location
- Wrap each glass container in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place the bubble-wrapped container into a 2.7-mil zip-type bag, removing trapped air.
- Place glass containers inside a polyethylene bottle filled with vermiculite; seal the bottle.
- Place sufficient amount of vermiculite in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- Place sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tieing or taping.
- The maximum volume of preserved solution in the cooler must not exceed 500 ml.
- The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

**Sulfuric Acid Solution UN2796** LTD. QTY.

- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix a Corrosive label to the outside of the cooler.
- Affix package orientation labels on two opposite sides of the cooler.
- Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of cooler labeling/marking locations is shown in Figure 1.

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**Note**: Samples containing less than the exception concentration of 0.35 percent H<sub>2</sub>SO<sub>4</sub> by weight will be shipped as nonregulated or nonhazardous in accordance with the procedure described in Section 1.4.

**Note**: No marking or labeling can be obscured by strapping or duct tape.

**Note**: The inner packaging of dangerous goods must be placed into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

- When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).
- Complete a Dangerous Goods Airbill.

## 7.0 Packaging and Shipping Limited-Quantity Radioactive Samples

7.1 Containers

The inner packaging containers that may be used for these shipments include:

Any size sample container

#### 7.2 Description/Responsibilities

- The qualified shipper will determine that the samples undergoing shipment contain no other contaminant that meets the definition of hazardous material as defined by DOT.
- The qualified shipper will ship all samples that meet the Class 7 definition of radioactive materials and meet the activity requirements specified in Table 7 of 49 CFR 173.425, as Radioactive Materials in Limited Quantity. The qualified shipper will verify that all packages and their contents meet the requirements of 49 CFR 173.421, *Limited Quantities of Radioactive Materials*.
- The packaging used for shipping will meet the general requirements for packaging and packages specified in 49 CFR 173.24 and the general design requirements provided in 173.410. These standards state that a package must be capable of withstanding the effects of any acceleration, vibration, or vibration resonance that may arise under normal condition of transport without any deterioration in the effectiveness of the closing devices on the various receptacles or in the integrity of the package as a whole and without loosening or unintentionally releasing the nuts, bolts, or other securing devices even after repeated use.
- If the shipment is from a DOE facility, radiological screenings will be completed on all samples taken. The qualified shipper will review the results of each screening (alpha, beta, and gamma speciation). Samples will not be shipped offsite until the radiological screening has been performed.
- The total activity for each package will not exceed the relevant limits listed in Table 7 of 49 CFR 173.425. The A₂ value of the material will be calculated based on all radionuclides found during previous investigations (if any) in the area from which the samples are derived. The A₂ values to be used will be the most restrictive of all potential radionuclides as listed in 49 CFR 173.435.
- The radiation level at any point on the external surface of the package bearing the sample(s) will not exceed 0.005 mSv/hour (0.5 mrem/hour). These will be verified by dose and activity monitoring before shipment of the package.
- The removable radioactive surface contamination on the external surface of the package will not exceed the limits specified in 49 CFR 173.443(a). CDM will apply the DOE-established free release criteria for removable surface contamination of less than 20 dpm/100 cm² (alpha) and 1,000 dpm/100 cm² (beta/gamma). It shall be noted that these values are more conservative than the DOT requirements for removable surface contamination.
- The qualified shipper will verify that the outside of the inner packaging is marked "Radioactive."
- The qualified shipper will verify that the excepted packages prepared for shipment under the provisions of 49 CFR 173.421 have a notice enclosed, or shown on the outside of the package, that reads, "This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910."

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**Note**: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

## 7.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Survey documentation/radiation screening results (if shipping from DOE or radiological sites)
- Orientation labels
- Excepted quantities label
- Consignor/consignee labels

## 7.4 Packaging of Limited-Quantity Radioactive Samples

The following steps are to be followed when packaging limited-quantity sample shipments:

- The cooler is to be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.
- Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.
- All sample containers will be properly labeled and the label protected with waterproof tape before sampling.
- At a minimum the label must contain:
  - Project name Sample location
  - Project number Sample identification number
  - Date and time of sample collection Collector's initials
- This step is optional; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.
- Place sufficient amount of vermiculite, or approved packaging material, in the bottom of the cooler to absorb any leakage that may occur.
- Place a garbage bag in the cooler.
- Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.
- If required, place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
- Seal the garbage bag by tieing or taping.
- Place a label marked Radioactive on the outside of the sealed bag.
- Enclose a notice that includes the name of the consignor or consignee and the following statement: "This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910."
- Note that both DOT and IATA apply different limits to the quantity in the inside packing and in the outside packing.
- The maximum weight of the package shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.
- Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.
- If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
- If a cooler is used, wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
- Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
- Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
- Affix package orientation labels on two opposite sides of the cooler/package.
- Affix a completed Excepted Quantities label to the side of the cooler/package.
- Secure any marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment.
- An example of the cooler labeling/marking is shown in Figure 2.

**Note**: No marking or labeling can be obscured by strapping or duct tape.



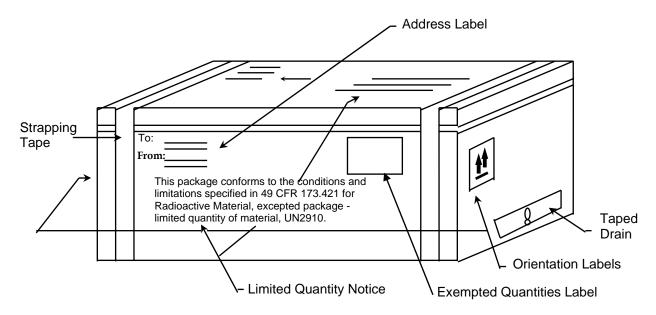
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Complete the Shipment Quality Assurance Checklist (Appendix B).

**Note**: Except as provided in 49 CFR 173.426, the package will not contain more than 15 grams of <sup>235</sup>U. **Note**: A declaration of dangerous goods is not required.

Figure 2
Radioactive Material – Limited-Quantity Cooler Marking Example



#### 8.0 References

U. S. Environmental Protection Agency. Region IV. February 1991 or current. *Standard Operating Procedures and Quality Assurance Manual*.

\_\_\_\_\_. 1996 or current. Sampler's Guide to the Contract Laboratory Program, EPA/540/R-96/032.

Title 49 Code of Federal Regulations, Department of Transportation. 2005 or current revision. *Hazardous Materials Table, Special Provisions, Hazardous, Materials Communications, Emergency Response Information, and Training Requirements*, 49 CFR 172.

Title 49 Code of Federal Regulations, Department of Transportation. 2005 or current revision. *Shippers General Requirements for Shipments and Packagings*, 49 CFR 173.

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## **Appendix A**

			Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity
Sam	ole Pa	ckaging	
Yes	No	N/A	
		0	The VOA vials are wrapped in bubble wrap and placed inside a zip-type bag. The VOA vials are placed into a polyethylene bottle, filled with vermiculite, and tightly sealed.
			The drain plug is taped inside and outside to ensure control of interior contents.
			The samples have been placed inside garbage bags with sufficient bags of ice to preserve samples at 4°C.
		0	The cooler weighs less than the 66-pound limit for limited-quantity shipment. The garbage bag has been sealed with tape (or tied) to prevent movement during shipment.
		<u> </u>	The chain-of-custody has been secured to the interior of the cooler lid.  The cooler lid and sides have been taped to ensure a seal.  The custody seals have been placed on both the front and back hinges of the cooler, using waterproof tape.
Air V	/aybill	Comple	etion
Yes	No	N/A	
			Section 1 has the shipper's name, company, and address; the account number, date, internal billing reference number; and the telephone number where the shipper can be reached.
			Section 2 has the recipient's name and company along with a telephone number where they can be reached.
			Section 3 has the <b>Bill Sender</b> box checked. Section 4 has the <b>Standard Overnight</b> box checked. Section 5 has the <b>Deliver Weekday</b> box checked. Section 6 has the number of packages and their weights filled out. Was the total of all
			packages and their weights figured up and added at the bottom of Section 6? Under the <b>Transport Details</b> box, the <b>Cargo Aircraft Only</b> box is obliterated, leaving only the <b>Passenger and Cargo Aircraft</b> box.
			Under the <b>Shipment Type</b> , the <b>Radioactive</b> box is obliterated, leaving only the <b>Non-Radioactive</b> box.
			Under the Nature and Quantity of Dangerous Goods box, the Proper Shipping Name, Class or Division, UN or ID No., Packing Group, Subsidiary Risk, Quantity and Type of Packing, Packing Instructions, and Authorization have been filled out for the type of chemical being sent.
			The Name, Place and Date, Signature, and Emergency Telephone Number appears at the bottom of the FedEx Airbill.
		П	The statement "In accordance with IATA/ICAO" appears in the <b>Additional Handling</b>

The Emergency Contact Information at the bottom of the FedEx Airbill is truly someone who can respond any time of the day or night.

**Information** box.

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Proper Shipping Name	Class or Division	UN or ID No.	Packing Group	Sub Risk	Quantity	Packing Instruction	Authorization
Hydrochloric Acid Solution	8	UN1789	II		1 plastic box × 0.5 L	Y809	Ltd. Qty.
Nitric Acid Solution (with less than 20%)	8	UN2031	II		1 plastic box × 0.5 L	Y807	Ltd. Qty.
Sodium Hydroxide Solution	8	UN1824	II		1 plastic box × 0.5 L	Y809	Ltd. Qty.
Sulfuric Acid Solution	8	UN2796	II		1 plastic box × 0.5 L	Y809	Ltd. Qty.
Methanol	3	UN1230	II		1 plastic box × 1 L	Y305	Ltd. Qty.

## Sample Cooler Labeling

Yes	No	N/A	
			The proper shipping name, UN number, and Ltd. Qty. appears on the shipping container
			The corresponding hazard labels are affixed on the shipping container; the labels are no obscured by tape.
			The name and address of the shipper and receiver appear on the top and side of the shipping container.
			The air waybill is attached to the top of the shipping container.
			<b>Up Arrows</b> have been attached to opposite sides of the shipping container.
			Packaging tape does not obscure markings or labeling.

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# Appendix B Shipment Quality Assurance Checklist

Date: _			Shipper:	Destination:			
Item(s)	Descr	iptio	n:				
Radion	uclide(	(s): _					
Radiolo	gical S	Surv	ey Results: surfacemrem/hr	1 meter			
Instrum	ent Us	sed:	Mfgr:	Model:			
S/N:				_ Cal Date:			
Yes	No		Limited-Quantity or Instrument	and Article			
		1.	Strong tight package (package that will not leak meto transportation).	naterial during conditions normally incidental			
		2.	Radiation levels at any point on the external surfamrem/hr.	ace of package less than or equal to 0.5			
		3.	Removable surface contamination less than 20 dy (beta/gamma).	pm/100 cm <sup>2</sup> (alpha) and 1,000 dpm/100 cm <sup>2</sup>			
			Outside inner package bears the marking "Radioa Package contains less than 15 grams of <sup>235</sup> U (che				
_	<u> </u>		Notice enclosed in or on the package that include statement, "This package conforms to the concern 173.421 for radioactive material, excepted UN2910."	es the consignor or consignee and the ditions and limitations specified in 49			
		7.	Activity less than that specified in 49 CFR 173.42 Package Quantity:	5. Permissible package limit:			
		8.	On all air shipments, the statement Radioactive I quantity of material shall be noted on the air way				
Qualifie	d Ship	per:	Signature:				

SOP 2-2 Revision: 5

Date: March 2007

Signature/Date

Prepared: Tim Eggert Technical Review: Matt Brookshire

E-Signed by Michael C. Malloy VERIFY authenticity with Approvel

QA Review: Jo Nell Mullins

E-Signed by P. Michael Schwan
VERIEY authenticity with ApproveIt

Issued:

Signature/Date

## 1.0 Objective

This standard operating procedure (SOP) presents guidance for the management of investigation-derived waste (IDW). The primary objectives for managing IDW during field activities include:

Approved:

- Leaving the site in no worse condition than existed before field activities
- Removing wastes that pose an immediate threat to human health or the environment
- Proper handling of onsite wastes that do not require offsite disposal or extended aboveground containerization
- Complying with federal, state, local, and facility applicable or relevant and appropriate requirements (ARARs)
- Careful planning and coordination of IDW management options
- Minimizing the quantity of IDW

## 2.0 Background

#### 2.1 Definitions

**Hazardous Waste** - Discarded material that is regulated listed waste, or waste that exhibits ignitability, corrosivity, reactivity, or toxicity as defined in 40 CFR 261.3 or state regulations.

**Investigation-Derived Wastes** - Discarded materials resulting from field activities such as sampling, surveying, drilling, excavations, and decontamination processes that, in present form, possess no inherent value or additional usefulness without treatment. Wastes may be solid, sludge, liquid, gaseous, or multiphase materials that may be classified as hazardous or nonhazardous.

Mixed Waste - Any material that has been classified as hazardous and radioactive.

Radioactive Wastes - Discarded materials that are contaminated with radioactive constituents with specific activities in concentrations greater than the latest regulatory criteria (i.e., 10 CFR 20).

**Treatment, Storage, and Disposal Facility (TSDF)** - Permitted facilities that accept hazardous waste shipments for further treatment, storage, and/or disposal. These facilities must be permitted by the U. S. Environmental Protection Agency (EPA) and appropriate state and local agencies.

#### 2.2 Discussion

Field investigation activities result in the generation of waste materials that may be characterized as hazardous or radioactive waste. IDWs may include drilling muds, cuttings, and purge water from test pit and well installation; purge water, soil, and other materials from collection of samples; residues from testing of treatment technologies and pump and treat systems; personal protective equipment (PPE); solutions (aqueous or otherwise) used to decontaminate nondisposable protective clothing and equipment; and other wastes or supplies used in sampling and testing potentially hazardous or radiologically contaminated material.

**Note**: The client's representatives may not be aware of all potential contaminants. The management of IDW must comply with applicable regulatory requirements.

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## 3.0 General Responsibilities

**Site Manager** - The site manager is responsible for ensuring that all IDW procedures are conducted in accordance with this SOP. The site manager is also responsible for ensuring that handling of IDW is in accordance with site-specific requirements.

**Project Manager** - The project manager is responsible for identifying site-specific requirements for the disposal of IDW in accordance with federal, state, and/or facility requirements.

**Field Crew Members** - Field crew members are responsible for implementing this SOP and communicating any unusual or unplanned condition to the project manager's attention.

**Note**: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/project specific quality assurance plan.

## 4.0 Required Equipment

Equipment required for IDW containment will vary according to site-specific/client requirements. Management decisions concerning the necessary equipment required shall consider: containment method, sampling, labeling, maneuvering, and storage (if applicable). Equipment must be onsite and inspected before commencing work.

#### 4.1 IDW Containment Devices

The appropriate containment device (drums, tanks, etc.) will depend on site- or client-specific requirements and the ultimate disposition of the IDW. Typical IDW containment devices can include:

- Plastic sheeting (polyethylene) with a minimum thickness of 20 millimeters
- Department of Transportation (DOT)-approved steel containers
- Polvethylene or steel bulk storage tanks

Containment of IDW shall be segregated by waste type (i.e., solid or liquid, corrosive or flammable, etc.) and source location. Volume of the appropriate containment device shall be site-specific.

#### 4.2 IDW Container Labeling

A "Waste Container" or "IDW Container" label or indelible marking shall be applied to each container. Labeling or marking requirements for onsite IDW not expected to be transported offsite are:

- Labels and markings that contain the following information: project name, generation date, location of waste origin, container identification number, sample number (if applicable), and contents (drill cuttings, purge water, PPE, etc.).
- Each label or marking will be applied to the upper one-third of the container at least twice, on opposite sides.
- Containers that are 5 gallons or less may only require one label or set of markings.
- Labels or markings will be positioned on a smooth part of the container. The label must not be affixed across container bungs, seams, ridges, or dents.
- Labels must be constructed of a weather-resistive material with markings made with a permanent marker or paint pen and capable of enduring the expected weather conditions. If markings are used, the color must be easily distinguishable from the drum color.
- Labels will be secured in a manner to ensure the label remains affixed to the container.

Labeling or marking requirements for IDW expected to be transported offsite must be in accordance with the requirements of 49 CFR 172.

#### **4.3 IDW Container Movement**

Staging areas for IDW containers shall be predetermined and in accordance with site-specific and/or client requirements. Arrangements shall be made before field mobilization as to the methods and personnel required to safely transport IDW containers to the staging area. Transportation offsite onto a public roadway is prohibited unless 49 CFR 172 requirements are met.

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#### 4.4 IDW Container Storage

Containerized IDW shall be staged pending chemical analysis or further onsite treatment. Staging areas and bulk storage procedures are to be determined according to site-specific requirements. Containers are to be stored in such a fashion that the labels can be easily read. A secondary/spill container must be provided for liquid IDW storage and as appropriate for solid IDW storage.

#### 5.0 Procedures

The three general options for managing IDW are (1) collection and onsite disposal, (2) collection for offsite disposal, and (3) collection and interim management. Attachment 1 summarizes media-specific information on generation processes and management options. The option selected shall take into account the following factors:

- Type (soil, sludge, liquid, debris), quantity, and source of IDW
- Risk posed by managing the IDW onsite
- Compliance with regulatory requirements
- IDW minimization and consistency with the IDW remedy and the site remedy

In all cases the client shall approve the plans for IDW. Formal plans for the management of IDW must be prepared as part of a work plan or separate document.

## 5.1 Collection and Onsite Disposal

#### 5.1.1 Soil/Sludge/Sediment

The options for handling soil/sludge/sediment IDW are as follows:

- 1. Return to boring, pit, or source immediately after generation as long as returning the media to these areas will not increase site risks (e.g., the contaminated soil will not be replaced at a greater depth than where it was originally so that it will not contaminate "clean" areas).
- 2. Spread around boring, pit, or source within the area of contamination (AOC) as long as returning the media to these areas will not increase site risks (e.g., direct contact with surficial contamination).
- 3. Consolidate in a pit within the AOC as long as returning the media to these areas will not increase site risks (e.g., the contaminated soil will not be replaced at a greater depth than where it was originally so that it will not contaminate "clean" areas).
- 4. Send to onsite TSDF may require analytical analysis before treatment/disposal.

**Note**: These options may require client and/or regulatory approval.

#### 5.1.2 Aqueous Liquids

The options for handling aqueous liquid IDW are as follows:

- 1. Discharge to surface water, only when IDW is not contaminated.
- Discharge to ground surface close to the well, only if soil contaminants will not be mobilized in the process and the action will not contaminate clean areas. If IDW from the sampling of background upgradient wells is not a community concern or associated with soil contamination, this presumably uncontaminated IDW may be released on the ground around the well.
- 3. Discharge to sanitary sewer, only when IDW is not contaminated.
- 4. Send to onsite TSDF may require analysis before treatment/disposal.

Note: These options may require analytical results to obtain client and/or regulatory approval.

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#### 5.1.3 Disposable PPE

The options for handling disposable PPE are as follows:

- Double-bag contents in nontransparent trash bags and place in onsite industrial dumpster, only if PPE is not contaminated.
- 2. Containerize, label, and send to onsite TSDF may require analysis before treatment/disposal.

## 5.2 Collection for Offsite Disposal

Before sending to an offsite TSDF, analysis may be required. Manifests are required. In some instances, a bill of lading can be used for nonhazardous solid IDW (i.e., wooden pallets, large quantities of plastic sheeting). Arrangements must be made with the client responsible for the site to sign as generator on any waste profile and all manifests or bill of ladings; it is CDM's policy not to sign manifests. The TSDF and transporter must be permitted for the respective wastes. Nonbulk containers (e.g., drums) must have a DOT-approved label adhered to the container and all required associated placard stickers before leaving for a TSDF off site. These labels must include information as required in 49 CFR 172. Bulk containers (i.e., rolloffs, tanks) do not require container specific labels for transporting off site, but must include appropriate placards as required in 49 CFR 172.

#### 5.2.1 Soil/Sludge/Sediment

When the final site remedy requires offsite treatment and disposal, the IDW may be stored (e.g., drummed, covered in a waste pile) or returned to its source until final disposal. The management option selected shall take into account the potential for increased risks, applicable regulations, and other relevant site-specific factors (e.g., weather, storage space, and public concern/perceptions).

#### 5.2.2 Aqueous Liquids

When the final site remedy requires offsite treatment and disposal, the IDW may be stored (e.g., mobile tanks or drums with appropriate secondary containment) until final disposal. The management option selected shall take into account the potential for increased risks, applicable regulations, and other relevant site-specific factors (e.g., weather, storage space, and public concern/perceptions).

#### 5.2.3 Disposable PPE

When the final site remedy requires offsite treatment disposal, the IDW may be containerized and stored. The management option selected shall take into account potential for increased risks, applicable regulations, and other relevant site-specific factors (e.g., weather, storage space, and public concern/perceptions).

#### 5.3 Collection and Interim Management

All interim measures must be approved by the client and regulatory agencies.

- 1. Storing IDW onsite until the final action may be practical in the following situations:
  - Returning wastes (especially sludges and soils) to their onsite source area would require reexcavation for disposal in the final remediation alternative.
  - Interim storage in containers may be necessary to provide adequate protection to human health and the environment.
  - Offsite disposal options may trigger land disposal regulations under the Resource Conservation and Recovery Act (RCRA). Storing IDW until the final disposal of all wastes from the site will eliminate the need to address this issue more than once.
  - Interim storage may be necessary to provide time for sampling and analysis.
- 2. Segregate and containerize all waste for future treatment and/or disposal.
  - Containment options for soil/sludge/sediment may include drums or covered waste piles in AOC.
  - Containment options for aqueous liquids may include mobile tanks or drums.
  - Containment options for PPE may include drums or roll-off boxes.

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### 6.0 Restrictions/Limitations

Site Managers Shall Determine the Most Appropriate Disposal Option for Aqueous Liquids on a Site-Specific Basis. Parameters to consider, especially when determining the level of protection, include the volume of IDW, the contaminants present in the groundwater, the presence of contaminants in the soil at the site, whether the groundwater or surface water is a drinking water supply, and whether the groundwater plume is contained or moving. Special disposal/handling may be needed for drilling fluids because they may contain significant solid components.

Disposable sampling materials, disposable PPE, decontamination fluids, etc. will always be managed on a site-specific basis. Under No Circumstances Shall These Types of Materials Be Brought Back to the Office or Warehouse.

#### 7.0 References

Environmental Resource Center. 1997. *Hazardous Waste Management Compliance Handbook 2nd Edition*. Karnofsky (Editor).

Academy of Certified Hazardous Materials Manager. May 1999. Hazardous Materials Management Desk Reference. Cox.

Title 49 Code of Federal Regulations, Department of Transportation. 2005 or current revision. *Hazardous Materials Table, Special Provisions, Hazardous, Materials Communications, Emergency Response Information, and Training Requirements*, 49 CFR 172.

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U. S. Environmental Protection Agency. 1987. A Compendium of Superfund Field Operations Methods, EPA/540/P-87/001.1.
August 1990. Low-Level Mixed Waste: A RCRA Perspective for NRC Licensees, EPA/530-SW-90-057.
May 1991. Management of Investigation-Derived Wastes During Site Inspections, EPA/540/G-91/009.
January 1992. Guide to Management of Investigation-Derived Wastes, 9345.3-03FS.
Region IV. November 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual.

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	Attachment 1							
		agement Options						
Type of IDW	Generation Processes	Management Options						
Soil	<ul> <li>Well/Test pit installations</li> <li>Borehole drilling</li> <li>Soil sampling</li> </ul>	Onsite Disposal  Return to boring, pit, or source immediately after generation  Spread around boring, pit, or source within the AOC  Consolidate in a pit (within the AOC)  Send to onsite TSDF  Offsite Disposal  Client to send to offsite TSDF  Interim Management  Store for future treatment and/or disposal						
Sludge/Sediment	■ Sludge pit/sediment sampling	Onsite Disposal  Return to boring, pit, or source immediately after generation  Send to onsite TSDF  Offsite Disposal  Client to send to offsite TSDF  Interim Management  Store for future treatment and/or disposal						
Aqueous Liquids (groundwater, surface water, drilling fluids, wastewaters)	<ul> <li>Well installation/development</li> <li>Well purging during sampling</li> <li>Groundwater discharge during pump tests</li> <li>Surface water sampling</li> <li>Wastewater sampling</li> </ul>	Onsite Disposal  Pour onto ground close to well (nonhazardous waste)  Discharge to sewer  Send to onsite TSDF  Offsite Disposal  Client to send to offsite commercial treatment unit  Client to send to publicly owned treatment works (POTW)  Interim Management  Store for future treatment and/or disposal						
Decontamination Fluids	<ul> <li>Decontamination of PPE and equipment</li> </ul>	Onsite Disposal  Send to onsite TSDF  Evaporate (for small amounts of low contamination organic fluids)  Discharge to ground surface  Offsite Disposal  Client to send to offsite TSDF  Discharge to sewer  Interim Management  Store for future treatment and/or disposal						
Disposable PPE and Sampling Equipment	<ul> <li>Sampling procedures or other onsite activities</li> </ul>	Onsite Disposal  Place in onsite industrial dumpster Send to onsite TSDF  Offsite Disposal Client to send to offsite TSDF  Interim Management Store for future treatment and/or disposal						

Adapted from U. S. Environmental Protection Agency, Guide to Management of Investigation-Derived Wastes, 9345-03FS, January 1992.

## Field Logbook Content and Control

**SOP 4-1** Revision: 6

RIFY authenticity

Date: March 2007

E-Signed by Michael C. Malloy

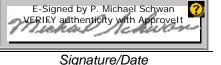
Prepared: Del Baird **Technical Review:** Laura Splichal

QA Review: Jo Nell Mullins

Approved:

Signature/Date

Issued:



## 1.0 Objective

The objective of this standard operating procedure (SOP) is to set CDM Federal (CDM) criteria for content entry and form of field logbooks. Field logbooks are an essential tool to document field activities for historical and legal purposes.

## 2.0 Background

#### 2.1 Definitions

Biota - The flora and fauna of a region.

Magnetic Declination Corrections - Compass adjustments to correct for the angle between magnetic north and geographical meridians.

#### 2.2 Discussion

Information recorded in field logbooks includes field team names; observations; data; calculations; date/time; weather; and description of the data collection activity, methods, instruments, and results. Additionally, the logbook may contain deviations from plans and descriptions of wastes, biota, geologic material, and site features including sketches, maps, or drawings as appropriate.

## 3.0 General Responsibilities

Field Team Leader (FTL) - The FTL is responsible for ensuring that the format and content of data entries are in accordance with this procedure.

Site Personnel - All CDM employees who make entries in field logbooks during onsite activities are required to read this procedure before engaging in this activity. The FTL will assign field logbooks to site personnel who will be responsible for their care and maintenance. Site personnel will return field logbooks to the records file at the end of the assignment.

**Note:** Responsibilities may vary from site to site. Therefore, all field team member responsibilities should be defined in the field plan or site-/project-specific quality assurance plan.

## 4.0 Required Equipment

- Site-specific plans
- Indelible black or blue ink pen
- Field logbook
- Ruler or similar scale

## 5.0 Procedures

#### 5.1 Preparation

In addition to this SOP, site personnel responsible for maintaining logbooks must be familiar with all procedures applicable to the field activity being performed. These procedures should be consulted as necessary to obtain specific information about equipment and supplies, health and safety, sample collection, packaging, decontamination, and documentation. These procedures should be located at the field office or vehicle for easy reference.

Field logbooks shall be bound with lined, consecutively numbered pages. All pages must be numbered before initial use of the logbook. Before use in the field, each logbook will be marked with a specific document control number issued by

## **Field Logbook Content and Control**

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the document control administrator, if required by the contract quality implementation plan (QIP). Not all contracts require document control numbers. The following information shall be recorded on the cover of the logbook:

- Field logbook document control number (if applicable).
- Activity (if the logbook is to be activity-specific), site name, and location.
- Name of CDM contact and phone number(s) (typically the project manager).
- Start date of entries.
- End date of entries.
- In specific cases, special logbooks may be required (e.g., waterproof paper for stormwater monitoring).

The first few (approximately five) pages of the logbook will be reserved for a table of contents (TOC). Mark the first page with the heading and enter the following:

#### **Table of Contents**

Date/Description (Start Date)/Reserved for TOC

Pages

The remaining pages of the table of contents will be designated as such with "TOC" written on the top center of each page. The table of contents should be completed as activities are completed and before placing the logbook in the records file.

## 5.2 Operation

Requirements that must be followed when using a logbook:

- Record work, observations, quantities of materials, calculations, drawings, and related information directly in the logbook. If data collection forms are specified by an activity-specific plan, this information does not need to be duplicated in the logbook. However, any forms used to record site information must be referenced in the logbook.
- Do not start a new page until the previous one is full or has been marked with a single diagonal line so that additional entries cannot be made. Use both sides of each page.
- Do not erase or blot out any entry at any time. Indicate any deletion by a single line through the material to be deleted. Initial and date each deletion. Take care to not obliterate what was written previously.
- Do not remove any pages from the book.

Specific requirements for field logbook entries include:

- Initial and date each page.
- Sign and date the final page of entries for each day.
- Initial and date all changes.
- Multiple authors must sign out the logbook by inserting the following:

Above notes authored by:

- (Sign name)
- (Print name)
- (Date)
- A new author must sign and print his/her name before additional entries are made.
- Draw a diagonal line through the remainder of the final page at the end of the day.
- Record the following information on a daily basis:
  - Date and time
  - Name of individual making entry
  - Names of field team and other persons onsite
  - Description of activity being conducted including station or location (i.e., well, boring, sampling location number) if appropriate
  - Weather conditions (i.e., temperature, cloud cover, precipitation, wind direction, and speed) and other pertinent data
  - Level of personal protection used
  - Serial numbers of instruments
  - Equipment calibration information
  - Serial/tracking numbers on documentation (e.g., carrier air bills)

## **Field Logbook Content and Control**

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Entries into the field logbook shall be preceded with the time (written in military units) of the observation. The time should be recorded frequently and at the point of events or measurements that are critical to the activity being logged. All measurements made and samples collected must be recorded unless they are documented by automatic methods (e.g., data logger) or on a separate form required by an operating procedure. In these cases, the logbook must reference the automatic data record or form.

At each station where a sample is collected or an observation or measurement made, a detailed description of the location of the station is required. Use a compass (include a reference to magnetic declination corrections), scale, or nearby survey markers, as appropriate. A sketch of station location may be warranted. All maps or sketches made in the logbook should have descriptions of the features shown and a direction indicator. It is preferred that maps and sketches be oriented so that north is toward the top of the page. Maps, sketches, figures, or data that will not fit on a logbook page should be referenced and attached to the logbook to prevent separation.

Other events and observations that should be recorded include:

- Changes in weather that impact field activities.
- Deviations from procedures outlined in any governing documents. Also record the reason for any noted deviation.
- Problems, downtime, or delays.
- Upgrade or downgrade of personal protection equipment.
- Visitors to the site.

#### 5.3 Post-Operation

To guard against loss of data as a result of damage or disappearance of logbooks, completed pages shall be periodically photocopied (weekly, at a minimum) and forwarded to the field or project office. Other field records shall be photocopied and submitted regularly and as promptly as possible to the office. When possible, electronic media such as disks and tapes should be copied and forwarded to the project office.

At the conclusion of each activity or phase of site work, the individual responsible for the logbook will ensure that all entries have been appropriately signed and dated and that corrections were made properly (single lines drawn through incorrect information, then initialed and dated). The completed logbook shall be submitted to the records file.

### 6.0 Restrictions/Limitations

Field logbooks constitute the official record of onsite technical work, investigations, and data collection activities. Their use, control, and ownership are restricted to activities pertaining to specific field operations carried out by CDM personnel and their subcontractors. They are documents that may be used in court to indicate dates, personnel, procedures, and techniques employed during site activities. Entries made in these logbooks should be factual, clear, precise, and nonsubjective. Field logbooks, and entries within, are not to be used for personal use.

#### 7.0 References

Sandia National Laboratories. 1991. Procedure for Preparing Sampling and Analysis Plan, Site-Specific Sampling Plan, and Field Operating Procedures, QA-02-03. Albuquerque Environmental Program, Department 3220, Albuquerque, New Mexico.

Sandia National Laboratories. 1992. *Field Operation Procedure for Field Logbook Content and Control*. Environmental Restoration Department, Division 7723, Albuquerque, New Mexico.

SOP 4-5 Revision: 7

Date: March 2007

E-Signed by Michael C. Malloy

Prepared: Steven Fundingsland Technical Review: Mike Higman

QA Review: Jo Nell Mullins

Approved:

ERIFY authenticity with Approve It

Signature/Date

Issued:

Signature/Date

E-Signed by P. Michael Schwan ERIFY authenticity with Approve It

## 1.0 Objective

The objective of this standard operating procedure (SOP) is to describe the general procedures required for decontamination of field equipment at nonradioactive sites. This SOP serves as a general guide and is applicable at most sites; however, it shall be noted that site-specific conditions (i.e., type of contamination, type of media sampled), the governing agency (e.g., EPA, DOE, USACE), and site-specific work plans, sampling and analysis plans and/or quality assurance (QA) project plans may require modifications to the decontamination procedures provided in this SOP. Decontamination of field equipment is necessary to ensure acceptable quality of samples by preventing cross contamination. Further, decontamination reduces health hazards and prevents the spread of contaminants offsite.

## 2.0 Background

#### 2.1 Definitions

Acid Rinse - A solution of 10 percent nitric or hydrochloric acid made from reagent grade acid and analyte-free water.

**Analyte-Free Water** - Tap water that has been treated so that the water contains no detectable heavy metals or other inorganic compounds. Analyte-free water shall be stored only in clean glass, stainless steel, or plastic containers that can be closed when not in use.

Clean - Free of contamination and when decontamination has been completed in accordance with this SOP.

**Cross Contamination** - The transfer of contaminants through equipment or personnel from the contamination source to less contaminated or noncontaminated samples or areas.

**Decontamination** - The process of rinsing or otherwise cleaning the surfaces of equipment to rid them of contaminants and to minimize the potential for cross contamination of samples or exposure of personnel.

**Material Safety Data Sheets (MSDS)** - These documents discuss the proper storage and physical and toxicological characteristics of a particular substance used during decontamination. These documents, generally included in site health and safety plans, shall be kept on site at all times during field operations.

**Organic-Free/Analyte-Free Water** - Tap water that has been treated so that the water meets the analyte-free water criteria and contains no detectable organic compounds. Organic-free/analyte-free water shall be stored only in clean glass, Teflon™, or stainless steel containers that can be closed when not in use.

**Potable Water** - Tap water may be obtained from any municipal system. Chemical analysis of the water source may be required before it is used.

**Sampling Equipment** - Equipment that comes into direct contact with the sample media. Such equipment includes split spoon samplers, well casing and screens, and spatulas or bowls used to homogenize samples.

**Soap** - Low-sudsing, nonphosphate detergent such as Liquinox™.

Solvent Rinse - Pesticide grade, or better, isopropanol, acetone, or methanol.

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#### 2.2 Associated Procedures

- CDM Federal SOP 1-1 Surface Water Sampling
- CDM Federal SOP 1-3 Surface Soil Sampling
- CDM Federal SOP 1-4 Subsurface Soil Sampling
- CDM Federal SOP 1-5 Groundwater Sampling Using Bailers
- CDM Federal SOP 1-7 Wipe Sampling
- CDM Federal SOP 1-9 Tap Water Sampling
- CDM Federal SOP 1-11 Sediment/Sludge Sampling
- CDM Federal SOP 2-2 Guide to Handling Investigation-Derived Waste
- CDM Federal SOP 3-1 Geoprobe® Sampling

## 3.0 Responsibilities

The project manager or designee, generally the field team leader (FTL), ensures that field personnel are trained in the performance of this procedure and that decontamination is conducted in accordance with this SOP and site-specific work plans. The FTL may also be required to collect and document rinsate samples (also known as equipment blanks) to provide quantitative verification that these procedures have been correctly implemented.

**Note:** Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific QA plan.

## 4.0 Required Equipment

- Stiff-bristle scrub brushes
- Plastic buckets and troughs
- Soap
- Nalgene or Teflon sprayers or wash bottles or 2- to 5-gallon, manual-pump sprayer (pump sprayer material must be compatible with the solution used)
- Plastic sheeting, plastic bags, and/or aluminum foil to keep decontaminated equipment clean between uses
- Disposable wipes, rags, or paper towels
- Potable water\*
- Analyte-free water
- Organic-free/analyte-free water
- Gloves, safety glasses, and other protective clothing as specified in the site-specific health and safety plan
- High-pressure pump with soap dispenser or steam-spray unit (for large equipment only)
- Appropriate decontamination solutions pesticide grade or better and traceable to a source (e.g., 10 percent and/or 1 percent nitric acid [HNO<sub>3</sub>], acetone, methanol, isopropanol, hexane)
- Tools for equipment assembly and disassembly (as required)
- 55-gallon drums or tanks for temporary storage of decontamination water (as required)
- Pallets for drums or tanks holding decontamination water (as required)

## 5.0 Procedures

All reusable equipment (nondedicated) used to collect, handle, or measure samples shall be decontaminated before coming into contact with any sampled media or personnel using the equipment. Decontamination of equipment shall occur either at a central decontamination station or at portable decontamination stations set up at the sampling location, drill site, or monitoring well location. The centrally located decontamination station shall include an appropriately sized bermed and lined area on which equipment decontamination shall occur and shall be equipped with a collection system and storage vessels. In certain circumstances, berming is not required when small quantities of water are being generated and for some short duration field activities (i.e., pre-remedial sampling). Equipment shall be transported to and from the decontamination station in a manner to prevent cross contamination of equipment and/or area. Precautions taken may include enclosing augers in plastic wrap while being transported on a flatbed truck.

<sup>\*</sup> Potable water may be required to be tested for contaminants before use. Check field plan for requirements.

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The decontamination area shall be constructed so that contaminated water is either collected directly into appropriate containers (5-gallon buckets or steel wash tubs) or within the berms of the decontamination area that then drains into a collection system. Water from the collection system shall be transferred into 55-gallon drums or portable tanks for temporary storage. Typically, decontamination water shall be staged until sampling results or waste characterization results are obtained and evaluated and the proper disposition of the waste is determined (SOP 2-2, *Guide to Handling Investigation-Derived Waste*). The exact procedure for decontamination waste disposal shall be discussed in the work plan. Also, solvent and acid rinse fluids may need to be segregated from other investigation-derived wastes.

All items that shall come into contact with potentially contaminated media shall be decontaminated before use and between sampling and/or drilling locations. If decontaminated items are not immediately used, they shall be covered either with clean plastic or aluminum foil depending on the size of the item. All decontamination procedures for the equipment being used are as follows:

#### **General Guidelines**

- Potable, analyte-free, and organic-free/analyte-free water shall be free of all contaminants of concern. Following the field QA sampling procedure described in the work plan, analytical data from the water source may be required.
- Sampling equipment that has come into contact with oil and grease shall be cleaned with methanol or other approved alternative to remove the oily material. This may be followed by a hexane rinse and then another methanol rinse. Regulatory or client requirements regarding solvent use shall be stated in the work plan.
- All solvents and acids shall be pesticide grade or better and traceable to a source. The corresponding lot numbers shall be recorded in the appropriate logbook.

**Note**: Solvents and acids are potentially hazardous materials and must be handled, stored, and transported accordingly. Solvents shall never be used in a closed building. See the site-specific health and safety plan and/or the chemical's MSDS for specific information regarding the safe use of the chemical.

- Decontaminated equipment shall be allowed to air dry before being used.
- Documentation of all cleaning and field QA sampling shall be recorded in the appropriate logbook.
- Gloves, boots, safety glasses, and any other personnel protective clothing and equipment shall be used as specified in the site-specific health and safety plan.

#### 5.1 Heavy Equipment Decontamination

Heavy equipment includes drilling rigs, well development rigs, and backhoes. Follow these steps when decontaminating this equipment:

- Establish a bermed decontamination area that is large enough to fully contain the equipment to be cleaned. If available, an existing wash pad or appropriate paved and bermed area may be used; otherwise, use one or more layers of heavy plastic sheeting to cover the ground surface and berms. All decontamination pads shall be upwind of the area under investigation.
- With the rig in place, spray areas (rear of rig or backhoe) exposed to contaminated media using a hot water high-pressure sprayer. Be sure to spray down all surfaces, including the undercarriage.
- Use brushes, soap, and potable water to remove dirt whenever necessary.
- Remove equipment from the decontamination pad and allow it to air dry before returning it to the work site.
- Record the equipment type, date, time, and method of decontamination in the appropriate logbook.

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After decontamination activities are completed, collect all contaminated wastewater, plastic sheeting, and disposable gloves, boots, and clothing in separate containers or receptacles. All receptacles containing contaminated items must be properly labeled for disposal as detailed in the field plan. Liquids and solids must be drummed separately.

### 5.2 Downhole Equipment Decontamination

Downhole equipment includes hollow-stem augers, drill pipes, rods, stems, etc. Follow these steps when decontaminating this equipment:

- Set up a centralized decontamination area, if possible. This area shall be set up to collect contaminated rinse waters and to minimize the spread of airborne spray.
- Set up a "clean" area upwind of the decontamination area to receive cleaned equipment for air-drying. At a minimum, clean plastic sheeting must be used to cover the ground, tables, or other surfaces on which decontaminated equipment is to be placed. All decontamination pads shall be upwind of any areas under investigation.
- Place the object to be cleaned on aluminum foil or plastic-covered wooden sawhorses or other supports. The objects to be cleaned shall be at least 2 feet above the ground to avoid splashback when decontaminating.
- Using soap and potable water in the hot water high-pressure sprayer (or steam unit), spray the contaminated equipment. Aim downward to avoid spraying outside the decontamination area. Be sure to spray inside corners and gaps especially well. Use a brush, if necessary, to dislodge dirt.
- If using soapy water, rinse the equipment using clean, potable water. If using hot water, the rinse step is not necessary if the hot water does not contain a detergent. If the hot water contains a detergent, this final clean water rinse is required.
- Using a suitable sprayer, rinse the equipment thoroughly with analyte-free water.
- Remove the equipment from the decontamination area and place in a clean area upwind to air dry.
- Record equipment type, date, time, and method of decontamination in the appropriate logbook.
- After decontamination activities are completed, collect all contaminated wastewaters, plastic sheeting, and disposable gloves, boots, and clothing in separate containers or receptacles. All receptacles containing contaminated items must be properly labeled for disposal. Liquids and solids must be drummed separately.

#### 5.3 Sampling Equipment Decontamination

Follow these steps when decontaminating sampling equipment:

- Set up a decontamination line on plastic sheeting. The decontamination line shall progress from "dirty" to "clean." A clean area shall be established upwind of the decontamination wash/rinse activities to dry the equipment. At a minimum, clean plastic sheeting must be used to cover the ground, table, or other surfaces that the decontaminated equipment is placed for drying.
- Disassemble any items that may trap contaminants internally. Do not reassemble the items until decontamination and air drying are complete.
- Wash the items with potable water and soap using a stiff brush as necessary to remove particulate matter and surface films. The items may be steam cleaned using soap and hot water as an alternative to brushing. *Note*: Polyvinyl chloride or plastic items shall not be steam cleaned. Items that have come into contact with concentrated and/or oily contaminants may need to be rinsed with a solvent such as hexane and allowed to air dry prior to this washing step.
- Thoroughly rinse the items with potable water.

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- If sampling for metals, thoroughly rinse the items with an acid solution (e.g., 10 percent nitric acid) followed by a rinse using analyte-free water. If sampling for organic compounds, thoroughly rinse the items with solvent (e.g., isopropanol) followed by a rinse using analyte-free water. The specific chemicals used for the acid rinse and solvent rinse phases shall be specified in the work plan. The acid rinsate and solvent rinsate must each be containerized separately. Acids and solvents are potentially hazardous materials and care must be exercised when using these chemicals to prevent adverse health affects (e.g., skin burns, irritation to the eyes and respiratory system). Appropriate personal protective equipment must be worn when using these chemicals. These chemicals (including spent rinsate) must be managed and stored appropriately. Special measures such as proper labels, paperwork, notification, etc. may be required when transporting or shipping these chemicals.
- Rinse the items thoroughly using organic-free/analyte-free water.
- Allow the items to air dry completely.
- After drying, reassemble the parts as necessary and wrap the items in clean plastic wrap or in aluminum foil.
- Record equipment type, date, time, and method of decontamination in the appropriate logbook.
- After decontamination activities are completed, collect all contaminated waters, used solvents and acids, plastic sheeting, and disposable personal protective equipment. Place the contaminated items in properly labeled drums for disposal. Liquids and solids must be drummed separately. Refer to site-specific plans for labeling and waste management requirements.

## 5.4 Pump Decontamination

Follow the manufacturer's recommendation for specified pump decontamination procedures. At a minimum, follow these steps when decontaminating pumps:

- Set up the decontamination area and separate "clean" storage area using plastic sheeting to cover the ground, tables, and other surfaces. Set up four containers: the first container shall contain dilute (nonfoaming) soapy water, the second container shall contain potable water, the third container shall be empty to receive wastewater, and the fourth container shall contain analyte-free water.
- The pump shall be set up in the same configuration as for sampling. Submerge the pump intake (or the pump, if submersible) and all downhole-wetted parts (tubing, piping, foot valve) in the soapy water of the first container. Place the discharge outlet in the wastewater container above the level of the wastewater. Pump soapy water through the pump assembly until it discharges to the waste container. Scrub the outside of the pump and other wetted parts with a metal brush.
- Move the pump assembly to the potable water container while leaving discharge outlet in the waste container. All downhole-wetted parts must be immersed in the potable water rinse. Pump potable water through the pump assembly until it runs clear.
- Move the pump intake to the analyte-free water container. Pump the water through the pump assembly. Pump the volume of water through the pump specified in the field plan. Usually, three pump-and-line-assembly volumes shall be required.
- Decontaminate the discharge outlet by hand, following the steps outlined in Section 5.3.
- Remove the decontaminated pump assembly to the clean area and allow it to air dry upwind of the decontamination area. Intake and outlet orifices shall be covered with aluminum foil to prevent the entry of airborne contaminants and particles.
- Record the equipment type, serial number, date, time, and method of decontamination in the appropriate logbook.

# Field Equipment Decontamination at Nonradioactive Sites

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#### 5.5 Instrument Probe Decontamination

Instrument probes used for field measurements such as pH meters, conductivity meters, etc. shall be decontaminated between samples and after use with analyte-free, or better, water.

### 5.6 Waste Disposal

Refer to site-specific plans and SOP 2-2 for waste disposal requirements. The following are guidelines for disposing of wastes:

- All wash water, rinse water, and decontamination solutions that have come in contact with contaminated equipment are to be handled, packaged, labeled, marked, stored, and disposed of as investigation-derived waste.
- Small quantities of decontamination solutions may be allowed to evaporate to dryness.
- If large quantities of used decontamination solutions shall be generated, each type of waste shall be contained in separate containers.
- Unless otherwise required, plastic sheeting and disposable protective clothing may be treated as solid, nonhazardous waste.
- Waste liquids shall be sampled, analyzed for contaminants of concern in accordance with disposal regulations, and disposed of accordingly.

## 6.0 Restrictions/Limitations

Nitric acid and polar solvent rinses are necessary only when sampling for metals or organics, respectively. These steps shall not be used, unless required, because of the potential for acid burns and ignitability hazards.

If the field equipment is not thoroughly rinsed and allowed to completely air dry before use, volatile organic residue, which interferes with the analysis, may be detected in the samples. The occurrence of residual organic solvents is often dependent on the time of year sampling is conducted. In the summer, volatilization is rapid, and in the winter, volatilization is slow. Check with your EPA region, state, and client for approved decontamination solvents.

#### 7.0 References

American Society for Testing and Materials. 2002. Standard Practice for Decontamination of Field Equipment at Nonradioactive Waste Sites, ASTM D5088-02. January 10.

Department of Energy. Hazardous Waste Remedial Actions Program. 1996. *Standard Operating Procedures for Site Characterization*, DOE/HWP-100/R1. September.

\_\_\_\_\_. Hazardous Waste Remedial Actions Program. 1996. *Quality Control Requirements for Field Methods*, DOE/HWP-69/R2. September.

U. S. Environmental Protection Agency. 1987. A Compendium of Superfund Field Operations Methods, EPA/540/P-87/001.1.

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\_\_\_. Region 2. 1989. CERCLA Quality Assurance Manual, Revision 1.

\_\_\_\_\_\_. Region 4. 2001. Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual. November.

**SOP 5-1** Revision: 8

Date: March 2007

Signature/Date

Prepared: Dave Johnson **Technical Review:** Steve Guthrie

> E-Signed by Michael C. Malloy Approved:

QA Review: Jo Nell Mullins

E-Signed by P. Michael Schwan ERIFY authenticity with Approvelt Signature/Date

Issued:

# 1.0 Objective

The objective of this standard operating procedure (SOP) is to establish the baseline requirements, procedures, and responsibilities inherent to the control and use of all measurement and test equipment (M&TE). Contractual obligations may require more specific or stringent requirements that must also be implemented.

## 2.0 Background

### 2.1 Definitions

Traceability - The ability to trace the history, application, or location of an item and like items or activities by means of recorded identification.

#### 2.2 Associated Procedures

- CDM Federal Technical SOP 4-1, Field Logbook Content and Control
- CDM Quality Procedures (QPs) 2.1 and 2.3
- Manufacturer's operating and maintenance and calibration procedures

#### 2.3 Discussion

M&TE may be government furnished (GF), rented or leased from an outside vendor, or purchased. It is essential that measurements and tests resulting from the use of this equipment be of the highest accountability and integrity. To facilitate that, the equipment shall be used in full understanding and compliance with the instructions and specifications included in the manufacturer's operations and maintenance and calibration procedures and in accordance with any other related project-specific requirements.

## 3.0 Responsibilities

All staff with responsibility for the direct control and/or use of M&TE are responsible for being knowledgeable of and understanding and implementing the requirements contained herein as well as any other related project-specific requirements.

The project manager (PM) or designee (equipment coordinator, quality assurance coordinator, field team leader, etc.) is responsible for initiating and tracking the requirements contained herein.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance plan.

## 4.0 Requirements for M&TE

- Determine and implement M&TE related project-specific requirements
- The maintenance and calibration procedures must be followed when using M&TE
- Obtain the maintenance and calibration procedures if they are missing or incomplete
- Attach or include the maintenance and calibration procedures with the M&TE
- Prepare and record maintenance and calibration in an equipment log or a field log as appropriate (Figure 1)
- Maintain M&TE records
- Label M&TE requiring routine or scheduled calibration (when required)
- Perform maintenance and calibration using the appropriate procedure and calibration standards
- Identify and take action on nonconforming M&TE

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## 5.0 Procedures

## 5.1 Determine if Other Related Project-Specific Requirements Apply

#### For all M&TE:

The PM or designee shall determine if M&TE related project-specific requirements apply. If M&TE related project-specific requirements apply, obtain a copy of them and review and implement as appropriate.

## 5.2 Obtain the Operating and Maintenance and Calibration Documents

#### For GF M&TE that is to be procured:

**Requisitioner** - Specify that the maintenance and calibration procedures be included.

### For GF M&TE that is acquired as a result of a property transfer:

Receiver - Inspect the M&TE to determine whether maintenance and calibration procedures are included with the item. If missing or incomplete, order the appropriate documentation from the manufacturer.

#### For M&TE that is to be rented or leased from an outside vendor:

Requisitioner - Specify that the maintenance and calibration procedures, the latest calibration record, and the calibration standards certification be included. If this information is not delivered with the M&TE, ask the procurement division to request it from the vendor.

## 5.3 Prepare and Record Maintenance and Calibration Records

#### For all M&TE:

PM or Designee - Record all maintenance and calibration events in a field log unless other project-specific requirements apply.

#### For GF M&TE only (does not apply to rented or leased M&TE):

If an equipment log is a project specific requirement, perform the following:

Receiver - Notify the PM or designee for the overall property control of the equipment upon receipt of an item of M&TE.

#### PM or Designee and User:

- Prepare a sequentially page numbered equipment log for the item using the maintenance and calibration form (or equivalent) (Figure 1).
- Record all maintenance and calibration events in an equipment log.

## 5.4 Label M&TE Requiring Calibration

#### For GF M&TE only (does not apply to rented or leased M&TE):

If calibration labeling is a project specific requirement, perform the following:

## PM or Designee:

- Read the maintenance and calibration procedures to determine the frequency of calibration required.
- If an M&TE item requires calibration before use, affix a label to the item stating "Calibrate Before Use."
- If an M&TE item requires calibration at other scheduled intervals, e.g., monthly, annually, etc., affix a label listing the date of the last calibration, the date the item is next due for a calibration, the initials of the person who performed the calibration, and a space for the initials of the person who shall perform the next calibration.

## 5.5 Operating, Maintaining or Calibrating an M&TE Item

#### For all M&TE:

PM or Designee and User - Operate, maintain, and calibrate M&TE in accordance with the maintenance and calibration procedures. Record maintenance and calibration actions in the equipment log or field log.

## 5.6 Shipment

#### For GF M&TE:

Shipper - Inspect the item to ensure that the maintenance and calibration procedures are attached to the shipping case, or included, and that a copy of the most recent equipment log entry page (if required) is included with the shipment. If the maintenance and calibration procedures and/or the current equipment log page (if required) is missing or incomplete, do not ship the item. Immediately contact the PM or designee and request a replacement.

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#### For M&TE that is rented or leased from an outside vendor:

**Shipper** - Inspect the item to ensure that the maintenance and calibration procedures and latest calibration and standards certification records are included prior to shipment. If any documentation is missing or incomplete, do not ship the item. Immediately contact the procurement division and request that they obtain the documentation from the vendor.

#### 5.7 Records Maintenance

#### For GF M&TE:

**PM or Designee** - Create a file upon the initial receipt of an item of M&TE or calibration standard. Organize the files by contract origin and by M&TE item and calibration standard. Store all files in a cabinet, file drawer, or other appropriate storage media at the pertinent warehouse or office location.

Receiver - Forward the original packing slip to the procurement division and a photocopy to the PM or designee.

## PM or Designee and User:

- Maintain all original documents in the equipment file except for the packing slip and field log.
- File the photocopy of the packing slip in the M&TE file.
- Record all maintenance and calibration in an equipment log or field log (as appropriate). File the completed equipment logs in the M&TE records. Forward completed field logs to the PM for inclusion in the project files.

#### For M&TE rented or leased from an outside vendor:

**Receiver** - Forward the packing slip to the procurement division.

#### User:

- Forward the completed field log to the PM for inclusion in the project files.
- Retain the most current maintenance and calibration record and calibration standards certifications with the M&TE item and forward previous versions to the PM for inclusion in the project files.

#### 5.8 Traceability of Calibration Standards

## For all items of M&TE:

## PM or Designee and User:

- When ordering calibration standards, request nationally recognized standards as specified or required. Request commercially available standards when not otherwise specified or required. Or, request standards in accordance with other related project-specific requirements.
- Require certifications for standards that clearly state the traceability.
- Require Material Safety Data Sheets to be provided with standards.
- Note standards that are perishable and consume or dispose of them on or before the expiration date.

#### 5.9 M&TE That Fails Calibration

#### For any M&TE item that cannot be calibrated or adjusted to perform accurately:

#### **PM or Designee**

- Immediately discontinue use and segregate the item from other equipment. Notify the appropriate PM and take appropriate action in accordance with the CDM QP 2.3 for nonconforming items.
- Review the current and previous maintenance and calibration records to determine if the validity of current or previous measurement and test results could have been affected and notify the appropriate PM(s) of the results of the review.

## 6.0 Restrictions/Limitations

On an item-by-item basis, exemptions from the requirements of this SOP may be granted by the Headquarters health and safety manager and/or Headquarters quality assurance director. All exemptions shall be documented by the grantor and included in the equipment records as appropriate.

## 7.0 References

CDM Federal Programs Corporation. 2007. Quality Assurance Manual. Rev. 11.

CDM Federal Programs Corporation. 2005. Government Property Manual. Rev. 3.

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Date: March 2007

# Figure 1

A subsidiary of Camp Dresser & McKee Inc.	Maintenance and Calibration
Date: Time: (a.m./p.m.)	
Employee Name:	Equipment Description:
Contract/Project:	Equipment ID No.:
Activity:	Equipment Serial No.:
Main	tenance
Maintenance Performed:	
Comments:	
Signature:	Date:
_	n/Field Check
Calibration Standard:	Concentration of Standard:
Lot No. of Calibration Standard:	Expiration Date of Calibration Standard:
Pre-Calibration Reading:	Post-Calibration Reading:
Additional Readings:	Additional Readings:
Additional Readings:	Additional Readings:
Pre-Field Check Reading:	Post-Field Check Reading:
Adjustment(s):	
Calibration:   Passed   Failed	
Comments:	
Signature:	Date:

# Site-Specific Sampling Guidance Libby Superfund Site

Guidance No.: CDM-LIBBY-05, Revision 2

Guidance Title: Soil Sample Collection at Residential and Commercial Properties

Approved by:	
[homps Class]	5/10/07
Technical Reviewer	Date
Textromell	5lidoz
QA Reviewer	Date
Mal C.M	05/10/07
Volpe Center Approval	/ / Date
	5/10/07
ELA Approval	/ Date

# Section 1 Purpose

The goal of this standard operating procedure (SOP) is to provide a consistent method for the collection of 30-point composite surface soil sampling to support all investigations conducted at the Libby Superfund Site and specified in governing guidance documents. This SOP describes the equipment and operations used for sampling surface soils in residential and commercial areas, which will be submitted for the analysis of Libby amphibole asbestos. Refer to each investigation-specific guidance documents or work plan for detailed modifications to this SOP, where applicable. The EPA Team Leader or their designate must approve deviations from the procedures outlined in this document prior to initiation of the sampling activity.

## **Section 2**

# Responsibilities

Successful execution of this SOP requires a clear hierarchy of assigned roles with different sets of responsibilities associated with each role. All staff with responsibility for the collection of soil samples is responsible for understanding and implementing the requirements contained herein as well as any other governing guidance documents.

Task Leader (TL) or Field Team Leader (FTL) - The TL or FTL is responsible for overseeing sample collection processes as described in EPA approved governing guidance documents (i.e., site-specific sampling and analysis plans [SAPs], quality assurance project plans [QAPPs], etc.). The TL or FTL is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and all governing guidance documents. The TL or FTL will communicate with the field team members regarding the specific collection objectives and anticipated situations that require deviation from this SOP. It is also the responsibility of the TL or FTL to communicate the need for any deviations from the SOP with the appropriate EPA personnel (team leader or their designate), and document the deviations using a Field Modification Form provided in each SAP or QAPP.

<u>Field team members</u> - Field team members performing the sampling described in this SOP are responsible for adhering to the applicable tasks outlined in this procedure while collecting samples at properties associated with the Libby Superfund Site. The field team members should have limited discretion with regard to collection procedures but should exercise judgment regarding the exact location of sample points, within the boundaries outlined by the TL or FTL.



# Section 3 Equipment

# ■ Measuring tape or wheel – Used to estimate the square footage of each land use

- Pin flags Used to identify composite points within each sampling area.
- <u>Trowel or push probe</u> For collecting surface soil samples.
- <u>Shovel</u> For collecting surface soil samples.
- <u>Stainless steel mixing bowl</u> Used to mix and homogenize composite soil samples after collection. Zip-top bags may also be used for homogenization if approved by the governing guidance documents.
- <u>Gloves</u> For personal protection and to prevent cross-contamination of samples (disposable, powderless plastic or latex).
- <u>Sample container</u> Gallon-sized zip-top plastic bags (2 per sample).
- <u>Field clothing and personal protective equipment (PPE)</u> As specified in the current version of the site health and safety plan (HASP).
- <u>Field sprayers</u> Used to suppress dust during sample collection and to decontaminate nondisposable sampling equipment between samples.
- <u>Deionized (DI) water</u>- Used in field sprayers to suppress dust and to clean and decontaminate sampling equipment.
- <u>Plastic bristle brush</u> Used to clean and decontaminate sampling equipment.
- Wipes Disposable, paper. Used to clean and decontaminate sampling equipment.
- <u>Aluminum foil</u> Used to wrap decontaminated sampling equipment in between uses to prevent contamination during transport.
- Alconox Used to clean and decontaminate sampling equipment weekly.
- 6-mil poly bag Used to store and dispose of investigation-derived waste (IDW).
- Trash bag Used to store and dispose of general trash.
- <u>Field logbook/PDA</u> Used to record progress of sampling effort and record any problems and field observations.



- <u>Visual Vermiculite Estimation Form (VVEF)</u> Used to record semi-quantitative estimates of visual vermiculite at each sub-sample location and point inspection (PI).
- Permanent marking pen Used to label sample containers.
- Sample ID Labels (Index IDs) Pre-printed stickers used to label sample containers.
- Cooler or other rigid container Used to store samples while in the field.
- <u>Custody Seals</u> For ensuring integrity of samples while in the field and during shipping.

# Section 4

# Sampling Approach

Upon arrival at each property, the field team will locate all parcels requiring sample collection depending on the investigation-specific objectives detailed in governing guidance documents. Parcels on a property will be sectioned into zones that share a similar land use. Zones established by land use areas may be subdivided based on site conditions (e.g., access, construction setup considerations, etc.). Use areas include:

- Specific Use Area (SUA): flowerbed, garden, flowerpot, stockpile, play area, dog pen, driveway (non-paved), parking lot (non-paved), road (non-paved), alley (non-paved)
- Common Use Area (CUA): yard, former garden, former flowerbed, walkway
- <u>Limited Use Area (LUA)</u>: pasture, maintained/mowed field, overgrown areas with trails/footpaths, overgrown areas in between SUAs/CUAs
- <u>Interior Surface Area (ISA)</u>: soil floor of garage, pumphouse, shed, crawlspace, earthen basement
- Non-Use Areas(NUA): wooded lot, un-maintained field. NUAs will be identified but will not be sampled at this time because they are not presently considered a complete exposure pathway. However, to the extent that NUAs may become a complete exposure pathway in the future, EPA may revisit NUAs at a later date.

After areas have been designated as zones (i.e., SUA zones, CUA zones, LUA zones, NUA zones, ISA zones), the field team will measure the zones with a measuring wheel and label the zone type and approximate square footage on the field sketch and/or design drawings. There is not a minimum or maximum square footage restriction on any zone.

In establishing zones at the property, no area type may be combined with any other area type. For example, driveways and flowerbeds are both SUAs but will be



separated into unique zones for soil sampling. Similarly, large CUAs such as yards may be subdivided into front yard, side yard, and back yard zones dependent on site conditions. Sectioning properties into additional zones will be at the discretion of the FTL but consistent among the teams. Conversely, not all land use areas previously mentioned will be applicable at every property.

It is anticipated that SUAs and ISA zones will generally tend to be smaller parcels. Combining small, proximal SUAs into one zone will be at the discretion of the FTL but consistent among teams. With the exception of proximal SUAs, all other land use areas will be contiguous when establishing zones at each property.

Composite sampling requires soil collection from multiple (sub-sample) points. Composite samples will be collected from similar land use areas (i.e., SUA, CUA, etc.) and will not be combined with any other use area. One composite sample will be collected from each zone that does not contain visual vermiculite.

For SUAs (e.g., driveway, garden, dog pen, etc.), composite samples will be collected from the 0- to 6-inch depth interval. If a depth of 6 in. cannot be attained given the varying levels of compaction in driveways, roads, etc. the maximum depth attainable will be documented in the field logbook/PDA. For non-SUAs (e.g., yard, former flowerbed, crawlspace, etc.), composite samples will be collected from 0 to 3 inches. All composite soil samples will have 30 sub-samples (i.e., 30-point composite sample) of approximately equal size for a final sample volume between 2,000 and 2,500 grams. Table 1 lists the sample depth for each type of land use area.

Table 1 Sampling Area and Depth						
Land Use Area	Label	Sampling Depth (Inches)				
Special Use Area	SUA	0 – 6				
Common Use Areas	CUA	0 – 3				
Limited Use Area	LUA	0 – 3				
Non-Use Area	NUA	Not Sampled				
Interior Surface Zone	IS	0 – 3				

As each sub-sample is collected, the soil will be inspected for visual vermiculite (VV) and the location and semi-quantitative estimates of VV will be recorded as prescribed in the SOP for Semi-Quantitative Visual Estimation of Vermiculite in Soil, Revision 1 (CDM 2007a).

Areas of SUAs with VV will not be sampled. Instead, the location will be recorded in the field logbook/PDA and on the field sketch or design drawing. If the SUA is of substantial size (greater than 1000 square feet [ft²]), and the VV is localized, additional PIs will be collected to determine the extent of VV and a sample will be collected from the remainder of the zone that does not contain VV. If the SUA measures less than 1,000 ft² and VV is present, a sample will not be collected from that SUA. Proximal



SUAs will not be combined into a SUA zone if VV is present. If visible vermiculite is not observed, proceed with sample collection of the SUA zone

# Section 5

# Sample Collection

Don the appropriate PPE as specified in the governing HASP. A new pair of disposable gloves is to be worn for each sample collected. Segregate land use areas on the property into zones as described in Section 4. To reduce dust generation during sampling, use a sprayer with DI water to wet each sub-sample location prior to collection. Use the trowel to check beneath the surface soil layer, but do not advance more than 6 inches. If VV is observed, record the information on the field sketch or design drawing. If VV is observed within a large SUA, do not collect a sample from the area containing VV as described above.

Within each zone, select 30 sub-sample locations equidistant from each other. These 30 sub-sample locations will comprise the 30-point composite sample for that zone. All composite sub-samples will originate from the same land use area. For example, do not mix sub-samples from SUAs with sub-samples from LUAs.

Clean the sub-sample locations of twigs, leaves, and other vegetative material that can be easily removed by hand. Using the trowel or push probe, excavate a hole in the soil approximately 2 inches in diameter and 6 inches deep for SUAs, or 3 inches deep for non-SUAs, while placing the excavated material directly inside the gallon-sized zip-top plastic bag. Repeat this step for each subsequent sub-sample until the appropriate number of composite sub-samples has been collected. As each subsample is collected, inspect the location for VV as prescribed in the SOP for Semi-Quantitative Visual Estimation of Vermiculite in Soil, Revision 1 (CDM 2007a).

Samples collected from zones measuring greater than 3,000 ft<sup>2</sup> will require additional PIs to inspect the soil for VV, but no more than 30 sub-samples will be collected from a zone for each composite sample. Samples collected from zones measuring less than 3,000 ft<sup>2</sup> will have the same number of sub-samples as PIs unless additional PIs are required to identify the extent of localized VV.

Homogenize the sample as required by governing guidance documents. Once the sample is homogenized, fill the zip-top plastic bag to 1/3rd full (approximately 2000 grams). Affix the sample index ID label to the inside of the bag and write the index ID number on the outside of the bag, or affix an additional label using clear packing tape. Sample index ID numbers will be assigned based on the investigation-specific guidance document. Double bag the sample and repeat the labeling process for the outer bag. Decontaminate equipment between composite samples as described in Section 8.

Repeat steps outlined above until all samples from a property have been collected.



Soil field duplicate samples will be collected at the rate specified in governing guidance documents. Field duplicate samples will be collected as samples co-located in the same zone. The duplicate will be collected from the same number of subsamples as the parent sample, but the sub-sample locations of the duplicate sample will be randomly located in the zone. The inspection for VV at each sub-sample location will follow the same protocol as referenced above. These samples will be independently collected with separate sampling equipment or with the original sampling equipment after it has been properly decontaminated. For tracking purposes, the parent/duplicate sample relationship will be recorded in accordance with sample documentation requirements stated in the governing guidance document. These samples will be used to determine the variability of sample results in a given land use area. These samples will not be used to determine variability in sampling techniques.

# Section 6 Site Cleanup

IDW will be managed as prescribed in Section 3.2.10 of the Site-wide QAPP [SWQAPP] (CDM 2007b) or other applicable governing guidance documents. In general, replace the soil plug with excess sample volume. The soil should be placed back into the hole and tamped down lightly. If sandy areas such as playgrounds are sampled, refilling the soil plug is not necessary.

Rinse water, the roots of vegetation removed during sampling, and any excess soil volume may be returned to the sampled area.

## Section 7

## **Documentation**

A field logbook/PDA will be maintained by each individual or team that is collecting samples as prescribed in Section 3.2.4 of the SWQAPP (CDM 2007b) or other applicable governing guidance documents. Guidance documents will detail conditions which require attention, but at a minimum the following information should be collected:

- Project name
- Title of governing documents
- Property address
- Date
- Time
- Team members



- Weather conditions
- PPE used
- Locations of any samples or sub-samples that could not be acquired
- Descriptions of any deviations to the SAP or SOP and the reason for the deviation
- Relinquishment of samples to project sample coordinator

Complete required documentation as detailed in applicable governing guidance documents.

## **Section 8**

## Quality Assurance/Quality Control

Quality control samples will include:

■ Field duplicates

Detailed information on QC sample collection and frequency is prescribed in Section 3.1.3.2 of the SWQAPP (CDM 2007b) or other applicable governing guidance documents.

## **Section 8**

## Decontamination

All sampling equipment must be decontaminated prior to reuse. Specific instructions on sample equipment decontamination are included in the applicable governing guidance documents. In general, the procedure to decontaminate all soil sampling equipment is outlined below:

- Remove all visible contamination with plastic brush
- Use DI water and plastic brush to wash each piece of equipment
- Remove excess water present on the equipment by shaking
- Use a paper towel to dry each piece of equipment
- Wrap dried equipment in aluminum foil

Once a week all soil sampling equipment will be cleaning using Alconox and DI water.

Spent wipes, gloves, aluminum foil, and PPE must be disposed of or stored properly as IDW, specified in Section 3.2.10 of the SWQAPP (CDM 2007b) or other applicable governing guidance documents.



# **Section 9**

# Sample Custody

Field sample custody and documentation will follow the requirements described in Section 3.2.11 of the SWQAPP (CDM 2007b) or other applicable governing guidance documents.

# Section 10 Glossary

<u>Governing guidance documents</u> - The written document that spells out the detailed site-specific procedures to be followed by the project leader and the field personnel for completing specific investigations. These documents will clearly indicate specific requirements for the implementation of this SOP.

<u>Libby Superfund Site</u> – The Libby Superfund Site contains all buildings and land within the boundaries of each operable unit (OU) of the site and illustrated on the most recent version of the OU boundary map.

<u>Sub-sample</u> - The actual location at which the sample is taken. The dimension of a sample point is 2 inches across by 3 inches deep (6 inches for SUAs).

<u>Composite Sampling</u> - A sample program in which multiple sample points are compiled together and submitted for analysis as a single sample.

<u>Land Use Area</u> - A section of property segregated by how the property owner uses the area. The area can be classified as a SUA, LUA, CUA, ISA, or NUA.

# Section 11 References

CDM. 2007a. Semi-Quantitative Visual Estimation of Vermiculite in Soils at Residential and Commercial Properties, Revision 1. CDM-LIBBY-06.

CDM. 2007b. Site-Wide Quality Assurance Project Plan. Draft in review.



# Site-Specific Sampling Guidance Libby Superfund Site

SOP No.: CDM-LIBBY-06, Revision 1

SOP Title: Semi-Quantitative Visual Estimation of Vermand Commercial Properties	iculite in Soils at Residentiai
Approved by:	5/0/07
Technical Reviewer	Date
Tenfromell	<i>5/10/07</i> Date
QA Reviewer	<b>Date</b>
What In-	05/10/07 Data
Volpe Center Approval	/ Date
	5/10/07
EPA/Approval	Date

# Section 1 Purpose

EPA will identify and delineate the extent of any visible vermiculite (VV) present in soils as part of all investigations conducted at the Libby Superfund Site and specified in governing guidance documents. The goal of this standard operating procedure (SOP) is to provide a consistent approach to identify and characterize any VV present in soils.

The semi-quantitative approach presented in this SOP for visually estimating VV in soil will be revised as required to optimize data collection as the sampling teams gain experience. This will be accomplished by expanding and/or improving this SOP, supporting pictorial standards, and additional electronic data acquisition efforts, as necessary.

# Section 2 Definitions

<u>Specific Use Area (SUA)</u> - Discrete exterior parcels on a property with a designated specific use. Due to the nature of activities typically carried out in SUAs, residents may be especially vulnerable to exposures when Libby amphibole asbestos (LA) contaminated soil becomes airborne. SUAs may be bare or covered with varying amounts of vegetation. SUAs include:

- Flower Pot
- Flowerbed
- Garden
- Stockpile
- Play Area
- Dog Pen
- Driveway (non-paved)
- Parking Lot (non-paved)
- Road (non-paved)
- Alley (non-paved)

<u>Common Use Area (CUA)</u> – Exterior parcels on a property with varied or generic use. CUAs may be bare or covered with varying amounts of vegetation. CUAs include:

- Walkway
- Yard (front, back, side, etc.)
- Former Garden
- Former Flowerbed



<u>Limited Use Area (LUA)</u> – Exterior parcels on a property that are accessed, utilized, and maintained on a very limited basis. LUAs may be bare or covered with varying amounts of vegetation. LUAs include:

- Pasture
- Maintained/Mowed Fields
- Underneath porches/decks¹
- Overgrown Areas (with trails/footpaths, or between SUAs/CUAs)

<u>Interior Surface Area (ISA)</u> – Interior soil surfaces of buildings such as garages, pumphouses, sheds, and crawlspaces.

<u>Non-Use Area (NUA)</u> - Exterior parcels on a property with no current use (e.g., areas that are un-maintained and not accessed). NUAs may be bare or covered with varying amounts of vegetation. NUAs include:

- Wooded Lots
- Un-maintained Fields

Since NUAs are not currently accessed, they are not presently considered a complete exposure pathway. As such, semi-quantitative visual estimates of vermiculite in soil will not be captured at this time. However, to the extent that NUAs may become a complete exposure pathway in the future, EPA may revisit these NUAs at a later date.

Zone<sup>2</sup> – Parcels on a property that share a similar land use or subdivisions of a land use area based on site conditions (e.g., access, construction setup considerations, etc.) or sampling requirements. No area type may be combined with any other area type. For example, driveways and flowerbeds are both SUAs but will be separated into unique zones for visual inspection. Similarly, large CUAs such as yards may be subdivided into front yard, side yard, and back yard zones dependent on site conditions. Sectioning properties into additional zones will be at the discretion of the field team leader but consistent among the teams.

It is anticipated that SUAs and ISA zones will generally tend to be smaller parcels. Combining small, proximal SUAs into one zone will be at the discretion of the field team leader but consistent among teams. No ISA will be combined with any other ISA for visual inspection. There is not a maximum square footage restriction on any zone.

<sup>&</sup>lt;sup>1</sup> The soils underneath porches and decks will be classified as LUAs depending on ground clearance and accessibility to homeowners and pets. If these areas are not accessible, they will be classified as NUAs.

<sup>2</sup> The restriction on the maximum square footage of SUA zones (1,000 ft2) and non-SUA zones (2, 500 ft2) was eliminated from the previous iteration of this SOP after the data were reviewed by EPA and determined to sufficiently characterize the presence of VV regardless of zone square footage. Additionally, this will allow the flexibility necessary for field teams to identify areas of zones most cost effectively for removal purposes.



<u>Point Inspection (PI)</u> – Used in SUA, CUA, LUA, and ISA zones. A PI is an intrusive visual inspection of the top portions of the soil at a randomly selected point within a zone. A PI consists of the active displacement of the surface soil with a small shovel and visual inspection of the displaced soil to determine if VV is present. If VV is observed during the PI, the location and a semi-quantitative estimate of VV contamination will be recorded.

# Section 3 Applicability

This SOP applies to properties within the Libby Superfund Site at varying stages of the removal process including, but not limited to, all screening and risk-based investigations, pre-design inspections, and removal actions. Investigation-specific modifications to this SOP are outlined in the governing guidance document for each investigation. The following locations on a property will be evaluated for the presence/absence of VV:

- All parcels on a property where soil samples are being collected.
- All parcels on a property where soil was non-detect for LA during previous sampling activities.
- All SUA parcels on a property that have not been previously characterized as containing VV

## **Section 4**

## **Procedure**

Figure 1 illustrates the procedures and decision rules for this SOP. The three primary procedural steps are listed below:

- Establish zones
- Perform PI
- Perform semi-quantification of visual vermiculite

Each is described in the following subsections.

## 4.1 Establish Zones

Upon arrival at the property, the field team will locate all areas requiring sample collection (i.e., where previous soil sample results were non-detect for LA or SUAs have not been previously characterized for VV). Parcels will be identified as SUA zones, CUA zones, LUA zones, NUA zones, or ISA zones. The field team will measure the zone sizes and note them on the field sketch and/or design drawings. Zones will be assigned according to the definitions provided above.



## 4.2 Point Inspections<sup>3</sup>

As defined above, a PI is an intrusive visual inspection performed for the entire surface of a zone. Professional judgment may be used to determine the exact location of PIs; however, the following guidelines will be implemented to maintain consistency.

A minimum of 30 PIs will be evaluated per zone if sampling is required within that zone. If soil sampling is not required, a minimum of 5 PIs will be evaluated within each zone. Zones larger than 500 square feet (ft²) will require evaluation at a minimum of 1 PI per 100 ft² (10 ft by 10 ft area). The PI locations will be randomly selected and will be spatially representative of the entire zone. Locations of the PIs and semi-quantitative estimates of VV (i.e., low, intermediate, or high) will be recorded on the field sketch for each PI. While a minimum of 5 PIs will be conducted per zone, there is no set maximum. Rather, the maximum number of PIs is variable—dependent upon the total area of the zone and achieving the minimum required frequency of 1 PI per 100 ft².

The following sections outline procedures for inspecting each use area (e.g., SUA, CUA, LUA, ISA). The procedure for semi-quantification of VV is provided in the next section.

#### SUA Zone:

- Visually inspect the PI point using a spade or trowel to remove any cover material, including excess debris (e.g., mulch, rock, etc.) and organic material, from the surface of the soil. Remove and visually inspect soil to a depth of 0-6 inches below ground surface<sup>4</sup>.
- If a depth of 6 in. cannot be attained given the varying levels of compaction in driveways, roads, etc. the maximum depth attainable will be documented in the field logbook.
- Record semi-quantitative estimate of VV observed as described in the following section.
- Replace soil and cover material.
- Repeat as necessary employing procedure outlined above.

## CUA and LUA Zones:

■ Visually inspect the PI point using a spade or trowel, carefully removing organic material, including grass, from the surface of the soil. Remove and visually inspect soil to a depth of 0 - 3 inches below ground surface<sup>5</sup>.

<sup>&</sup>lt;sup>4</sup> A soil depth of 6 inches for SUAs was chosen to approximate the depths to which digging would be expected during typical activities occurring in these SUA zones (e.g., gardening, child digging in dirt, etc.)
<sup>5</sup> A soil depth of 0-3 inches was chosen to approximate the depths to which soil disturbance would be most likely during typical activities occurring in these CUA and LUA zones (e.g., lawn mowing, etc.)



<sup>&</sup>lt;sup>3</sup> Surface Inspections- The non-intrusive visual inspection of the immediate surface of a zone was eliminated from the previous iteration of this SOP after their data were reviewed and determined by EPA to provide no additional information over that gained through Point Inspections.

- Record semi-quantitative estimate of VV observed as described in the following section.
- Carefully replace all soil and organic material.
- Repeat as necessary employing procedure outlined above.

## ISA Zone:

- Move items as necessary to access the soil surface.
- Visually inspect the PI points using a spade or trowel, remove and visually inspect soil to a depth of 0 3 inches below ground surface<sup>6</sup>.
- Record semi-quantitative estimate of VV observed as described in the following section.
- Repeat as necessary employing procedure outlined above.

If during the PI, VV is observed to be localized within a zone, the portion with vermiculite will be denoted on the field sketch. If additional PIs are necessary to determine the boundaries of the area, approximately 10 to 20% additional PIs will be evaluated to determine the extent of localized vermiculite.

## 4.3 Semi-Quantification of Visual Vermiculite

During PI, the field team will estimate the quantity of vermiculite observed. Each PI location for all zones will be assigned a semi-quantitative estimate of visible vermiculite content using a 4-point scale: none (blank), low (L), intermediate (M), and high (H)<sup>7</sup>. For PI locations where VV is observed, semi-quantitative estimates (e.g., L, M, or H) will be recorded on the field sketch. PI locations where VV is not observed will not be recorded on the field sketch. Photographs illustrating these quantities are attached to this SOP as Figure 2. Additionally, jars of vermiculite-containing soils representing these three levels will be available for training and reference.

Under the current version of this SOP, there will be no effort to design an approach to combine vermiculite levels for PIs within or among zones. While the viability of combining semi-quantitative visual estimates within or among zones may be assessed as a pilot-scale evaluation, any PI with visible vermiculite qualifies as vermiculite-containing soil for the area represented by the inspection point or inspection zone.

<sup>&</sup>lt;sup>7</sup> Based on EPA's review of previous data, the 5-level scale VV identification scheme was not meaningful and will be reduced to a 4-level scale. As such the quantity of "Gross" VV in the previous iteration of this SOP was combined with High. Previously collected data of Gross VV should be considered analogous to High VV under this revised SOP.



<sup>&</sup>lt;sup>6</sup> A soil depth of 0-3 inches was chosen to approximate the depths to which soil disturbance would be most likely during typical activities occurring in these IS zones (e.g., entering crawlspace, retrieving items from shed, etc.)

## Section 5

# **Health & Safety/Engineering Controls**

All personnel will carry out visual inspections in accord with proper personal protective equipment (PPE) and other monitoring/governing requirements outlined in the most recent version of the Site Health and Safety Plan governing the work being conducted.

All visual inspections will employ appropriate engineering controls to minimize dust (e.g., wetting soil during inspection) as prescribed in the Site-Specific Standard Operating Procedure for Soil Sample Collection (CDM-LIBBY-05, Revision 2).

## Section 6

# **Equipment Decontamination**

Equipment decontamination is not required between each PI from the same zone, but is required before moving to another inspection zone. Decontamination of equipment will be conducted as required by the governing guidance documents.

## Section 7

## **Documentation**

As noted above, information about the presence of vermiculite will be recorded on the field sketch or design drawing for the property under investigation. Each zone will be marked with:

- Zone type (i.e., SUA, CUA, LUA, NUA, or ISA)
- Zone area in ft²
- PI locations/points
- Semi-quantitative estimate of VV content for each PI (i.e., L, M, H)

In addition to field sketch/design drawing documentation, each field team will generate a Visual Vermiculite Estimation Form (VVEF) (Figure 3) to document the semi-quantitative visual estimates of VV for each PI for possible future information use. This form will be managed according to governing guidance documents.

# Section 8 Training

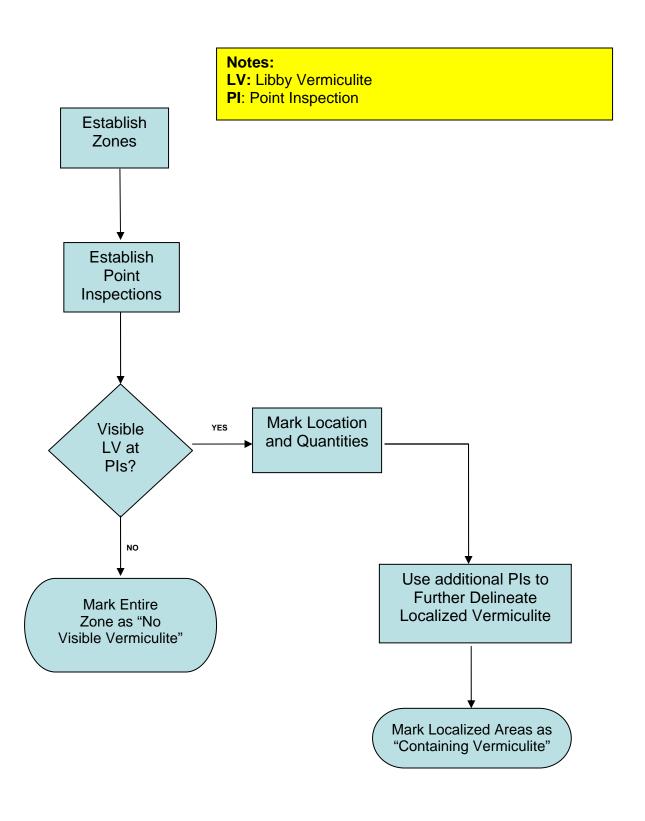
Every effort will be made to ensure consistency in the semi-quantitative evaluation of VV in soil to the extent possible. This will include training (e.g., field calibration), specimen examples (i.e., jars/photographs of low, intermediate, and high quantities of vermiculite, etc.), designated field staff, and oversight by the field team leader. Figures illustrating none, low, intermediate, and high quantities of vermiculite are attached to this SOP for reference (Figure 2).



To ensure consistency over time, the field team leader will verify semi-quantitative assignments at a rate of one property per team per week. The field team leader will sign off on those field sketches that were verified. If inconsistencies are noted, the field team leader will hold re-training with all teams participating simultaneously. Updates to the SOP and its attached specimen examples will occur as necessary and the EPA Project Team Leader and Technical Assistance Unit will be notified when these updates are recommended by the field team leader or field investigation manager.



Figure 1 – Visible Vermiculite Inspection Process



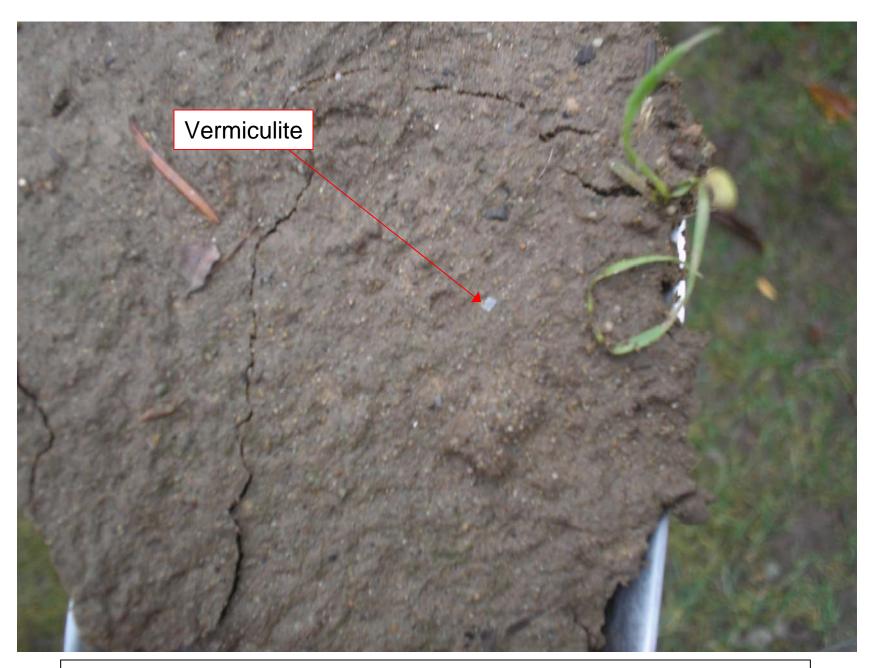


Figure 2a: Low Visible Vermiculite – A maximum of a few flakes of vermiculite observed within a given visual inspection point



Figure 2b: Intermediate Visible Vermiculite – Vermiculite easily observed throughout visual inspection point, including the surface.



Figure 2c: Intermediate Visible Vermiculite – Vermiculite easily observed throughout visual inspection point, including the surface.



Figure 2d: High Visible Vermiculite – Vermiculite easily observed throughout visual inspection point, including the surface.

# LIBBY SUPERFUND SITE Visual Vermiculite Estimation Form (VVEF)

Field Logbook No.:	Page No.:	Site Visit Date:	BD Number:
Address:		Structure Description: Property	
Occupant:	Phone No.		<u></u>
Owner (If different than occupant):	Phone No.		
Investigation Team:	Investigation Name		
Field Form Check Completed by (100% of Forms):	Visu	al Verification by Field Team Leader (10% of forms	3):

		Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6	Zone 7	Zone 8
Ty (SUA/CU	<b>/pe</b> A/LUA/IS)								
Desci	ription								
Area (squar	n Size re feet)								
	Comment er, etc.)								
H=High)	х								
<b>s</b> ntermediate,	L								
<b>PIS</b> (X=None, L=Low, M=Intermediate, H=High)	М								
(X=None,	н								
	Total	0	0	0	0	0	0	0	0

Areas previously identified for removal not inspected for visible vermiculite?	Yes	No	NA	Location(s):	
				-	
				-	

Page 1 of :

# Project-Specific Standard Operating Procedure Libby Asbestos Project

SOP No.: CDM-LIBBY-09, Revision 2
SOP Title: Global Positioning Satellite (GPS) Coordinate Collection and File Transfer Process
Project: Libby Asbestos Project
Project No.: 2616
Client: U.S. Department of Transportation (DOT)/Volpe Center

Authored by:

Date: 7/27/09

Date: 7/27/09

Terry Crowell

CDM Quality Assurance Reviewer

## 1.0 Objective

The objective of this standard operating procedure (SOP) is to provide a standardized approach for the collection and handling of GPS data at the Libby Asbestos Site (site).

# 2.0 Background

## 2.1 Definitions

**LibbySampling\_090615.ddf Data Dictionary** – All Trimble handheld units used at the site are pre-programmed with the LibbySampling data dictionary, specific to the spatial data collection needs for the Libby Asbestos Project. All personnel required to collect GPS data will be familiar with the contents of the LibbySampling data dictionary, which contains the following features: Sample, Building Location, Interest Point, and Interest Area. The Trimble units also are loaded with a generic data dictionary that handles collection of generic lines, points, and areas.

## 2.2 Discussion

The following attributes are required to be collected, as indicated in Table 1, for each feature type when a GPS coordinate is collected:

Table 1 – Attributes Collected in the LibbySampling_090615 Data Dictionary				
Feature Attributes Collected				
Sample	IndexID, LocationID, Comment			
Building Location	LocationID, Address, Comment			
Interest Point	Location, Land_Use, Comment			
Interest Area	Location, Land_Use, Comment			

These attributes are discussed in detail in Section 4 of this document.

## 3.0 Responsibilities

GPS data is collected by field staff as specified in the guidance document (e.g., sampling and analysis plan) governing the field work. Transfer of GPS data from the field equipment to the onsite server and transmittal of data off-site will be performed by designated administrative support staff. Documentation regarding off-site processing is posted on the Libby eRoom at <a href="https://team.cdm.com/eRoom/R8-RAC/Libby/0\_290a">https://team.cdm.com/eRoom/R8-RAC/Libby/0\_290a</a>.

## 4.0 Procedures

The following sections describe how GPS points are collected and handled for features commonly used at the site.

## 4.1 GPS Point Collection

The Sample Feature from the LibbySampling data dictionary is used to collect GPS points for sample locations.

### Soil Samples

For **Grab** samples, a GPS point is collected at the exact sampling location. Location IDs beginning with the prefix "SP" (indicating a sample point), are used for such locations.

For **Composite** samples, a GPS point is collected at the approximate center of each sample area. In the case of an irregular-shaped sample area or sample area that is non-continuous (e.g., a flowerbed that wraps around a house), a GPS point is collected at the center of the largest continuous sample area. Location IDs beginning with the prefix "SP" are used for such locations.

### Outdoor Stationary Air and Dustfall (Settled Dust) Samples

For permanent (i.e., samples representing a consistent monitoring zone or area collected on a routine schedule) outdoor stationary air and dustfall sample locations, a GPS point is collected at each unique sample location. All subsequent samples taken at that location will be assigned the same Location ID and X,Y coordinates. The GPS point is only collected once. Location IDs beginning with the prefix "SP" (indicating a sample point), are used for such locations.

GPS points are **not** collected for the following features, unless otherwise specified in the governing document:

- Stationary air, dust, and soil samples collected inside or beneath 4-sided structures (locations are associated with the X,Y coordinate of the building where the sample was collected)
- Stationary air samples, with the exception of permanent monitoring locations as designated in site-specific removal work plans or Response Action Work Plan Addenda
- Duplicate or Replicate air or dust samples (which are assigned the same Location ID and X,Y coordinates as the parent sample)
- Soil samples taken at depth from the same sample area as a previously-collected sample. The atdepth soil sample will be assigned the same Location ID as the shallower sample in order to relate both samples to the same X,Y coordinate.
- Duplicate or split soil samples (which are assigned the same Location ID and X,Y coordinates as the parent sample)
- Personal air samples (locations are associated with the X,Y coordinate of the building (i.e., BD Location ID) or property (i.e., AD Location ID) where the sample was collected)

## **Building Locations**

The Building Location Feature from the LibbySampling data dictionary is used to collect GPS points for building locations. For building locations, a GPS point is collected near the front door or main entrance of the building. Location IDs beginning with the prefix "BD" (indicating a building point), are used for such locations.

#### **Interest Point, Interest Area**

GPS points for these features are not routinely collected on the Libby Asbestos Project. However, they are included in the LibbySampling data dictionary in the event that a GPS point is collected for an area where no sampling is conducted, or a series of points is collected to document the perimeter of an interest area or sample area.

### **Pre-determined Sample Areas**

For pre-determined sample areas (e.g., gridded) where waypoints are available, the Trimble units may be pre-loaded with waypoint files to guide samplers to sampling locations. Pre-loading of coordinates is typically performed by a member of the Libby information management system team or by the field team leader. It should be noted that, in order to ensure GPS coordinate data are included in the project database, GPS points will also be collected at the time of sampling for sample locations located using waypoint files.

## 4.2 Operation of Trimble Handheld Units:

Operators must be standing at the sample location *before* the unit starts to collect positions. Once the unit has started collecting positions, the operator must remain standing at the sample location until the minimum required positions have been collected. A minimum of **30** positions will be collected for each GPS location. More positions will be required in circumstances where the position dilution of precision (PDOP) is greater than the default setting of 4.5.

## **Record-keeping Requirements:**

Serial numbers of the Trimble datalogger, receiver, and antenna will be recorded in a field logbook. GPS filenames will be recorded in the logbook. Recording GPS filenames on field sample data sheets (FSDSs) is not required.

## **Upgrades to Trimble Equipment and Software**

Trimble equipment and software is subject to change according to availability. The field team leader or designated administrative support staff is responsible for contacting the technical support of the vendor if there are any questions regarding setup, operation, or data transfer of models not covered below.

#### **Data Collection Instructions for Trimble Pro XRS:**

- Turn on the Trimble unit
- Select **Data Collection** from the main menu
- Select **Create New File** and press **Enter**. A generic default file name that begins with "*RO…*" followed by the date will appear.
- Name the file using the following naming convention: T1A10209, where T1 refers to the specific Trimble unit being used, A refers to the first file of the day (B would be the second file of the day, and so on), and 10219 refers to the date (October 21, 2009). The file name is limited to 8 characters on some units; therefore, the date notation must be MMDDY.
- Make sure the data dictionary is set to LibbySampling.
- Press **Enter** to bring up the **Start feature** menu.
- Arrow to the feature to be collected (i.e., Sample or Building Location). Press Enter

- Press the **F1** key to pause the unit until data collection can begin. (Note that if the unit is not paused, data collection will begin immediately).
- Enter the **Index ID** and **Location ID** exactly as they appear on the printed labels assigned to the sample.

### Index IDs, and Location IDs must match field documentation.

- Capitalize the ID prefixes where they are capitalized
- Include dashes where they are present
- Remove extra spaces

Data entry errors will prevent the coordinate data from exporting and validating correctly. Enter the property address and/or other information in the **Comment**. If required by the governing document, enter any additional information such as Owner, Sample Grid, Sample Location, etc. in the **Comment** field.

- Press the F1 key to **resume** collecting positions. The unit will beep for every position it collects, and display the total number of positions in the lower right corner.
- After the counter has reached the desired number of positions (30 positions), press **Enter** and then **Enter** to confirm and save your data point.
- Repeat this process for every new sample location.

Data may be viewed and edited by pressing **F2** (**Review**) from the **Start feature** menu, using the directional pad to scroll through the locations and pressing **Enter** to view the sample information. If edits are made to the data, be sure to press **Enter**. To exit without changing the data press **Esc**. Press **F2** (**New**) to return to the **Start feature** menu.

## Additional handheld features:

- **Review feature** allows for quick review/editing of keyed data
- **Repeat feature** use of this feature is not advised because of the likelihood to miss an edit of the index or location id fields. Points that have not been edited correctly will be rejected as duplicates when they are uploaded.
- Offset reduces the extra time associated with trying to capture GPS data under bridges, large trees, porches, facades and awnings, or while standing close to a building or other object that can deflect satellites signals from the GPS receiver.
- **Delete Feature** allows for deleting a feature from a file if, for example, no positions were collected or the sample is voided. This will prevent having to rectify data later on.
- **Rename File** allows for file name browsing/editing. This will prevent having to rectify data if done *before* the files are downloaded.
- **Delete File** allows for deleting a file from the handheld when necessary. This will prevent having to rectify data if it is done *before* the files are downloaded.

#### **Data Collection Instructions for Trimble GeoXT:**

- Turn on the unit and using the stylus, select **GPS** from the lower right menu. This will open the Terra Sync software.
- Wait for the GPS status screen to recognize at least 4 satellites. Depending on location, this can take several minutes and must be complete or data will not successfully be collected. The connected satellite names will appear on the left side of the screen highlighted to indicate a connection.

- Select **Data** from the upper left drop down menu. Use the file naming convention described above to create a file. Make sure the data dictionary is **LibbySampling**. Select **Create**.
- Confirm the antennae height by selecting Ok.
- Highlight the appropriate feature name and select **Create**. The unit will begin logging the point automatically. Enter the attribute data using the stylus and the keyboard icon located at the bottom of the touch screen. When recording is complete, select **Ok**, which saves the file and location information.
- To collect other points within the same feature file, select the **Options** menu then select **Repeat**.

# 4.3 GPS Data Transfer from Handheld Units to Libbysvr02

## GPS File Transfer to Libbysvr02 from Trimble Pro XRS

- Turn on the Trimble Unit
- *The unit will try to connect to the GPS receiver* press the **Esc** button
- Select File Manager
- Select **File Transfer** currently the data consists of .ssf files and is transferred to Libbysvr02\libbycommon\Data Management\Pfdata\Libby the file is named with an 8 character identifier: T + TrimbleUnitNo + file number (A for first file collected that day) + MMDDY
- Open Pathfinder Office
- Select Utilities
- Select Data Transfer
- Select Add
- Select **Datafile** Pathfinder will search for a connection to the Trimble Unit
- Connect the cable from the computer to the Trimble Unit
- A list of files will appear when the connection is complete
- Select Open
- Select Transfer All
- When the download is complete, close the data transfer window *if downloading files from several units, close and reopen this window between downloads*
- Delete files from the Trimble Unit all of the files will be listed double check that all the files were transferred to libbysvr02 before deleting

## GPS File Transfer to Libbysvr02 from Trimble Pro GeoXT

The Trimble GeoXT connects to a PC through the charger unit using a USB cable (*type A to type B*), and Microsoft Active Sync software. (*There are Active Sync connection settings to enable or disable once the device is connected to the PC. From the Active Sync menu, select Tools, select Options. These connect the Trimble to other Windows applications on the PC [e.g.; email, task managers, etc.]. The main reason to disable these settings at the Libby office is that the Trimble Units are shared and it does not make sense to activate them.)* 

- Turn on the Trimble Unit
- Select GPS from lower right corner (This opens up the TerraSync GPS software.)
- Select Setup
- Select Options
- Select Disconnect from GPS

- Select Data
- At the bottom of list, select File Manager
- Open Pathfinder
- Select Utilities
- Select Data Transfer
- From the Device list, select GIS Datalogger on Windows CE
- Click on the connect icon (the button with the checkmark circled in green). A picture on the right will indicate the connection status.

## 4.4 Transfer of GPS Data Off-site for Validation and Post-Processing

Following the download of files from the Trimble units, a copy of each file is made and filed in <code>Libbysvr02 \ libbycommon \ Data Management \ Pfdata \ Libby \ RawFiles</code>. The raw files are not modified but kept as the only copy of the original downloaded data files. The files are zipped and sent off-site for validation and post-processing. The .zip files are moved to <code>Libbysvr02 \ libbycommon \ Data Management \ Pfdata \ Libby \ QC and sent zip files</code>.

For reference on using Pathfinder export and ARCMap attribute tables see e-Rroom: Libby GIS folder: GPS to GIS procedure posted by Mike Schultz on August 29, 2006.

## 4.5 Equipment, Software & Configuration

For Trimble Pro XRS or Trimble GeoXT:

Software used

**for data transfer:** GPS Pathfinder Office 2.90 and 3.10

TerraSync

Software used

**for on-site QC**: GPS Pathfinder Office 2.90 and 3.10

ArcGIS ArcMap Microsoft Excel eLASTIC

#### **Configuration Settings (TSC1 5.27 software)**

Software can vary with rental equipment. Some settings can be changed to accommodate data collection needs.

Table - 2 Configuration Settings for Trimble Pro XRS					
GPS Rover Options - Logging C	)ptions				
Logging Intervals	Point feature	1 s			
	Line / area	3 s			
	Not in feature	none			
	Velocity	none			
Confirm end feature	no				
Minimum Positions	30				
Carrier phase	Carrier mode off				
	Minimum time	10mins			
GPS Rover Options – Position Filters					
Position mode	Manual 3D				

SNR mask	Elevation mask	15 degrees						
DOP type								
PDOP mask								
DODP switch		_						
CPS Rover Options - Real-time input								
Preferred correction source								
CPS Rover Options - General real-time settings								
Ornection age limit								
GPS Rover Options - Antenna options								
Height								
Measure								
Confirm         Never           Type         auto-filled when part number is entered           Part number         get part number off of antenna           GPS Rover Options – Initial Position         USft           North         USft           East         USft           GPS Rover Options – 2D altitude         Altitude(MSL)           Altitude(MSL)         USft           Computed at         time           Computed at         date           GPS Base Station Options – Logging Options         Logging Intervals           Logging Intervals         Measurements         5s           Audible Click         Yes           Log DOP data         Yes           GPS Base Station Options – Position Filters           Position made         Manual 3D           Elevation mask         15 degrees           SNR mask         4.0           PDOP mask         6.0           PDOP switch         4.0           GPS Base Station Options – Real-time output options           Real-time output mode         Gr           Radio type         Custom           Bud rate         9600           Data bits         8           Station         1 <td< td=""><td></td><td></td><td></td></td<>								
Type   auto-filled when part number is entered   get part number off of antenna   get part number off off   get part number of								
Part number			entered					
GPS Rover Options - Initial Position   North								
North								
East								
GPS Rover Options – 2D altitude								
Altitude(MSL)								
Computed at Computed at         time date           GPS Base Station Options – Logging Options         Logging Intervals           Loging Intervals         Measurements         5s           Audible Click         Yes         Jose Station Options – Position Filters           Log DOP data         Yes         Jose Station Options – Position Filters           Position mode         Manual 3D         Levation mask         Log								
Computed at GPS Base Station Options — Logging Options         5s           Logging Intervals         Measurements         5s           Positions         30s           Audible Click         Yes           Log DOP data         Yes           GPS Base Station Options — Position Filters           Position mode         Manual 3D           Elevation mask         15 degrees           SNR mask         4.0           PDOP switch         4.0           GPS Base Station Options — Real-time output options           Real-time output mode         off           Radio type         Custom           Baud rate         9600           Data bits         8           Stop bits         1           Parity         Odd           RTCM options         Station         1           Message type         Type 1           Message suffix         None           CTS flow control         Off           CTS xmit delay         0ms           RTS edge delay         0ms           GPS Base Station Options — Reference position           Datum         NAD 1983 (Conus)           Zone         11 North           NMEA/TSIP Output options <td></td> <td></td> <td></td>								
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Log DOP data		Positions	30s					
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SNR mask		sition Filters						
SNR mask								
PDOP mask	Elevation mask	15 degrees						
PDOP switch         4.0           GPS Base Station Options – Real-time output options           Real-time output mode         off           Radio type         Custom           Baud rate         9600           Data bits         8           Stop bits         1           Parity         Odd           RTCM options         Station         1           Message type         Type 1           Message interval         5s           Message suffix         None           CTS flow control         Off           CTS mit delay         0ms           RTS mode         High           RTS edge delay         0ms           GPS Base Station Options – Reference position           Datum         NAD 1983 (Conus)           Zone         11 North           NMEA/TSIP Output options           Output         TSIP           Baud rate         38400	SNR mask	4.0						
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Data bits         8           Stop bits         1           Parity         Odd           RTCM options         Station         1           Message type         Type 1           Message interval         5s           Message suffix         None           CTS flow control         Off           CTS xmit delay         0ms           RTS mode         High           RTS edge delay         0ms           GPS Base Station Options - Reference position           Datum         NAD 1983 (Conus)           Zone         11 North           NMEA/TSIP Output options           Output         TSIP           Baud rate         38400	Radio type	Custom						
Stop bits         1           Parity         Odd           RTCM options         Station         1           Message type         Type 1           Message interval         5s           Message suffix         None           CTS flow control         Off           CTS xmit delay         0ms           RTS mode         High           RTS edge delay         0ms           GPS Base Station Options - Reference position           Datum         NAD 1983 (Conus)           Zone         11 North           NMEA/TSIP Output options           Output         TSIP           Baud rate         38400	Baud rate	9600						
Parity         Odd           RTCM options         Station         1           Message type         Type 1           Message interval         5s           Message suffix         None           CTS flow control         Off           CTS xmit delay         0ms           RTS mode         High           RTS edge delay         0ms           GPS Base Station Options - Reference position           Datum         NAD 1983 (Conus)           Zone         11 North           NMEA/TSIP Output options           Output         TSIP           Baud rate         38400	Data bits	8						
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Message type         Type 1           Message interval         5s           Message suffix         None           CTS flow control         Off           CTS xmit delay         0ms           RTS mode         High           RTS edge delay         0ms           GPS Base Station Options – Reference position           Datum         NAD 1983 (Conus)           Zone         11 North           NMEA/TSIP Output options           Output         TSIP           Baud rate         38400	Parity	Odd						
Message interval         5s           Message suffix         None           CTS flow control         Off           CTS xmit delay         0ms           RTS mode         High           RTS edge delay         0ms           GPS Base Station Options - Reference position           Datum         NAD 1983 (Conus)           Zone         11 North           NMEA/TSIP Output options         TSIP           Baud rate         38400	RTCM options							
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CTS xmit delay		Message suffix	None					
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Datum         NAD 1983 (Conus)           Zone         11 North           NMEA/TSIP Output options         TSIP           Baud rate         38400								
Zone 11 North  NMEA/TSIP Output options Output TSIP  Baud rate 38400	GPS Base Station Options - Re							
NMEA/TSIP Output options Output TSIP Baud rate 38400								
Output TSIP Baud rate 38400		11 North						
Baud rate 38400								
Coordinate System UTM								
	Coordinate System UTM							

Map display options	All show with no background			
Units and Display				
Units	Distance(2D)	US Survey Ft		
	Area	Square feet		
	Velocity	Miles/Hour		
	Angle format	DDMMSSss		
	Order	North/East		
	North reference	True		
	Magnetic declination	Auto		
	Null string			
	Language	English		
Time and Date	24 hour clock	Yes		
	Time	##:##:##		
	Date format	MM/DD/YYYY		
	Date	MM/DD/YY weekday		
Quickmarks	Attributes	Repeat		
	Confirm	No		
Hardware(TSC1) software version 5.27				

Table 3 LibbySampling_090615 Data Dictionary
"LibbySampling_090615", Dictionary
"Sample", point, "", 1, seconds, 1, Code
"IndexID", text, 30, required, required, Label2
"LocationID", text, 30, required, required, SP-, Label1
"Comment", text, 30, normal, normal
"Building Location", point, "", 1, seconds, 1, Code
"LocationID", text, 30, required, required, BD-, Label1
"Address", text, 50, required, normal, Label2
"Comment", text, 30, normal, normal
"Interest Point", point, "", 1, seconds, 1, Code
"Location", text, 30, required, required, Label1
"Land_Use", text, 30, required, required, Label2
"Comment", text, 30, normal, normal
"Interest Area", area, "", 3, seconds, Code
"Location", text, 30, required, required, Label1
"Land_Use", text, 30, required, required, Label2
"Comment", text, 30, normal, normal

# **APPENDIX B Field Planning Meeting Form**

DOIN
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### **Field Planning Meeting Form**

Instructions: Prior to the field planning meeting (FPM), this form must be reviewed and approved by the project QA Coordinator and H&S Coordinator. This completed form must be placed in the project file with a DCN.

Meeting Date/Time:
Conducted By:
Project Name:
Project No.:
Project Manager:
Field Team Leader/Site Manager:
Field Activity Dates/Schedule:
Type of Field Event (SI, RI/FS, etc.):
List of Documents to be discussed/present at the FPM (H&S Plan, SAP, QAPP, CDAP, etc.):

### Attendees:

	Name (Printed)	Role	Affiliation	Signature
1		Field Team Leader		
2		QA Coordinator		
3		H&S Coordinator		
4		Sample Coordinator		
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				

Agenda: (Detail each item accordingly; attach additional sheets as needed.)				
F	Project Objectives:			
F	Field Measurements:			
T	Type and Number of Samples Planned to	be Collected (quantify all th	at apply):	
	Soil: Sediment: Microvacuum Dust: Other:	Dustfall: Personal Air: Stationary Air:	Groundwater: Surface Water: Bulk Materials:	
Þ	Analytical Method(s):			
(	QC Sample Type(s)/Number Required:			
E	Equipment/Calibration Standards Needec	<b>d</b> :		
F	Procedures to Follow (SOPs, etc.):			
T	raining Requirements:			
(	Other QA/QC Issues:			
Не	alth & Safety:			
ŀ	Health and Safety Action Levels:			
T	arget Contaminants and Highest Levels [	Detected:		
F	Personal Protective Equipment:			
(	Other Health and Safety Issues:			
Ag	enda Approvals:			
(	QA Coordinator (Signature and Date):			
H	Health and Safety Coordinator (Signature	and Date):		
CC:	Project File QA Coordinator			

**H&S** Coordinator

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# **APPENDIX C Inspection Forms**

# LIBBY ASBESTOS SITE Occupant Information Form (OIF)

General Information					
Address:					
Property ID:					
Location ID:					
Location Type:					
Location Description:					
Survey Date (Investigation Date):					
Event ID (Investigation Name):	SI	DI	SI/DI	ABS	ERS
Field Logbook Number:					
Logbook Page Numbers:					
Surveyors (Investigation Team Members):					
Field Form Check (100% of forms):					

Occupant Information					
Is there any knowledge of former miners, close relatives of miners, or any highly exposed persons living or visiting the property?	Yes	No			
Is the resident, past or present, diagnosed with an asbestos-related disease?	Yes	No			
	N/A				
Number of adult residents or employees?					
Number of child residents?					
Ago yango of child recidents?	0 - 6	7 - 12			
Age range of child residents?	13 - 18				
Donatha ann an Aideathan ann an Aideathan an	Yes	No			
Does the current resident have any outdoor pets?	N/A				

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General Information					
Address:					
Property ID:					
Location ID:					
Location Type:					
Location Description:					
Survey Date (Investigation Date):					
Event ID (Investigation Name):	SI	DI	SI/DI	ABS	ERS
Field Logbook Number:					
Logbook Page Numbers:					
Surveyors (Investigation Team Members):					
Field Form Check (100% of forms):					
Screening Field Check (2% of forms):					

\*Circle all that apply

Building Attributes					
Year of construction					
	Wood/Coal	Propane/Gas			
Heating source	Electric	None			
	Forced Air	Radiant			
Heat distribution	N/A				
	Air Conditioner	Swamp Cooler			
Cooling system	None				

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Attic Attributes		
	Finished	Unfinished
Type of Attic	Combined	N/A
Are there any entryway, porch, walkway awnings, or other	Yes	No
additions that share airspace with the main attic or each other?		N/A
Are there any entryway, porch, walkway awnings, or other	Yes	No
additions that do not share airspace with the main attic or each other?		N/A
Assistance in according to the state of the	Yes	No
Are there kneewalls present? (finished attics only)		N/A
	Yes	No
Is there flooring in the attic (above joist)?		N/A
	Yes	No
Are there any drop ceilings below the attic?		N/A
	Yes (note on sketch)	No
Are there any physical/structural issues, damages, or concerns?	(	N/A
	Electrical	Limited/Blocked access
Type of hazards near the attic access? (note on sketch)		None
Attic Inspection		
Vermiculite in attic?	Yes	No
		N/A
Vermiculite in attic-above-attic? (finished attics only)	Yes	No
vermeance in action above action (imission actions only)		N/A
Vermiculite under floor?	Yes	No
vermiculte under 11001:		N/A
Vermiculite on drop ceiling?	Yes	No
vermicance on drop cennig:		N/A
Manada Bashakiad Laga alka (6 t L. L. 19 L. 19	Yes	No
Vermiculite behind kneewalls? (finished attics only)		N/A

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Living Space Assessment					
Is vermiculite leaking into any living spaces?	Yes (note on sketch)	No			
	N/A				
Is vermiculite exposed in any cracks, holes, fixtures, etc.?	Yes (note on sketch)	No			
	N/A				
Is vermiculite exposed in any closets or cabinets?	Yes (note on sketch)	No			
	N/A				
Vermiculite present in interior walls	Yes	No			
	N/A				
Vermiculite present in exterior walls	Yes	No			
verificance present in exterior wans	N/A				
Understructure Attributes and Inspect	ion				
Type of Understructure	Basement	Crawlspace			
	Cellar	None			
	Concrete	Wood			
Type of flooring in understructure	Finished Flooring	Soil			
	N/A				
Are any areas of the understructure inaccessible?	Yes (note on sketch)	No			
Are any areas of the understructure maccessible:	N/A				
A	Yes (note on sketch)	No			
Are any areas of the understructure frequently accessed?	N/A				
Voyaniquiita in undaratuustuus?	Yes	No			
Vermiculite in understructure?	N/A				
Is vermiculity leaking into the understructure from above?	Yes (note on sketch)	No			
Is vermiculite leaking into the understructure from above?	N/A				

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Bulk/Building Materials			
Evidence of vermiculite additives used in building materials?	Yes	No	
	Ground Floor	Second Floor	
Location of building materials containg vermiculite	Third Floor	Attic	
	Understructure	N/A	
Are any vermiculite containing building materials friable?	Yes (note on sketch and collect sample)	No	
Are any vermiculte containing building materials mables	N/A		
Additional Information			
Is there any knowledge of the interior ever having vermiculite insulation?	Yes	No	
is there any knowledge of the interior ever having verificultie insulation:	N/A		

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	Gen	eral Informa	ation		
Address:					
Property ID:					
Survey Date (Investigation Date):					
Event ID (Investigation Name):	SI	DI	SI/DI	ABS	ERS
Field Logbook Number:					
Logbook Page Numbers:					
Surveyor (Investigation Team Members):					
Field Form Check (100% of forms):					
Screening Field Check (2% of forms):					

\*Circle all that apply

*Circle all that apply			
Secondary Structures			
Complete an IPIF for each secondary structure on the p	roperty		
Contamination beneath secondary structures?	Yes	No	
	Inaccessible	N/A	
If contamination is present beneath any secondary structures,	Yes	No	
are they mobile?	N//	Α	
Driveways and Walkways			
	Concrete	Asphalt	
Type of Driveway	Gravel	Soil	
	None		
Evidence of contamination extending beneath driveway(s)?	Yes (note on sketch)	No	
(only applicable for hard-surface driveways)	N/A		
	Concrete	Asphalt	
Type of walkways	Flagstone	Pavers	
	Gravel	None	
Evidence of contamination extending beneath walkway(s)?	Yes (note on sketch)	No	
(only applicable for hard-surface walkways)	N/A		
Additional Info			
Any special excavation concerns present? (indicate on sketch)	Yes	No	
Excessive household/personal items within areas requiring removal?	Yes	No	

### LIBBY ASBESTOS SITE Visual Vermiculite Estimation Form (VVEF)

Address:		
Property ID:		
Vermiculite Date (Inspection Date):		
Event ID (Investigation Name):		
Field Logbook Number:		
Page Number:		
Vermiculite Observer (Investi	gation Team Members):	
Field Form Check Comple	ted by (100% of Forms):	
Visual Verification by Field Team	Leader (10% of forms):	

Land	ID								
Locat	ion iD								
Location Type (e.g. SUA/CUA/LUA/ISA/EA)									
Location D	escription								
Locatio (squar									
Location (Cover	Comment r, etc.)								
Inspection (incl	Top Depth								
Inspection B (incl	ottom Depth nes)								
ite_Low, ulite_High)	х								
ne, L=Vermicul tte, H = Vermic	L								
PIs (X=Vermiculite_None, L=Vermiculite_Low, M=Vermiculite_Intermediate, H = Vermiculite_High)	М								
PIs (X=V M=Vermicul	Н								
	Total	0	0	0	0	0	0	0	0

### APPENDIX D Example Field Sample Data Sheets

Charge No.: 2616	
(write in or place label here)	

### LIBBY FIELD SAMPLE DATA SHEET (FSDS) FOR BULK MATERIALS

Sheet No.: <u>B- **000000**</u>

Address:		Sampling D	ate:
		Field Logbo	ok No:
Sampling Team: CDI	M Other Names	:	
Data Item	Sample 1	Sample 2	Sample 3
Index ID			
Location ID			
Location ib			
0 1 0			
Sample Group			
Location Description			
Category (circle)	FS	FS	FS
	Other	Other	Other
Matrix Type (circle)	Insulation	Insulation	Insulation
	Other	Other	Other
Sample Time			
Field Comments			
QC (Field Team)	Volpe:	Volpe:	Volpe:
Entered (LFO)	Entered Validated	Entered Validated	Entered Validated

#### v 090413

For Field Team Completion (Initials)	Completed by:	For Data Entry Entered b	y:
For eFSDS validation	Validated	Validated	Validated

Charge No.: 2616	
(write in or place label here)	

For eFSDS validation

Validated \_

### LIBBY FIELD SAMPLE DATA SHEET (FSDS) FOR SOIL

Sheet No.: <u>S- **000000**</u>

Address:		Field Logboo	ate: ok No:
Sampling Team: CDM	1 Other Names:_		
Data Item	Sample 1	Sample 2	Sample 3
Index ID			
Location ID			
Sample Group			
Location Description (circle)	Back yard Front yard Side yard Driveway Other	Back yard Front yard Side yard Driveway Other	Back yard Front yard Side yard Driveway Other
Category (circle)	FS FD of EB LB	FS FD of EB LB	FS FD of EB LB
Matrix Type (Surface soil unless other wise noted)	Surface Soil Other	Surface Soil Other	Surface Soil Other
Type (circle)	Grab # subsamples = 0 Comp. # subsamples	Grab # subsamples = 0 Comp. # subsamples	Grab # subsamples = 0 Comp. # subsamples
Sample Time			
Top Depth (inches below ground surface)			
Bottom Depth (inches below ground surface)			
Field Comments			
(Note if vermiculite was not observed in sample. For 30-point composites, note total # of visual inspection points of low (L), intermediate (M), or high (H) levels of vermiculite observed)	☐ no vermiculite observed L: M: H:	☐ no vermiculite observed L: M: H:	☐ no vermiculite observed L: M: H:
GPS File (fill in or circle)	Filename: NA	Filename: NA	Filename: NA
v 090526			
For Field Team Completion (Initials)	Completed by: QC by:		by:

Validated \_

Validated \_

### APPENDIX E Chain-of-Custody Record

### **Libby Asbestos Investigation**

Chain of Custody Record
Environmental Protection Agency, Region VIII 1595 Wynkoop Street Denver, Colorado 80202

	NO. <u>100030</u>	
Send to:		
	via:   hand delivery   shipped	

Sample Placed in Cooler/Bag	Index ID	Sample Date	Sample Time	Sample Matrix (S=Soil; W=Water; D=Dust; A=Air; B=Bulk Insulation)	Sample Type (G=Grab; C=Composite)	Volume (L) or Area (cm²)	Filter Pore Size (0.8µm or.45µm)	Analysis Request*	Comments	Sample Received by Lab

Total Number of Samples		END OF SUBMITTAL		
Additional Comments:				
Relinquished by (Signature and Company)	Date/Time	Received by (Signature and Company)	Date/Time	Sample Condition upon Receipt
Relinquished by (Signature and Company)	Date/Time	Received by (Signature and Company)	Date/Time	Sample Condition upon Receipt
Relinquished by (Signature and Company)	Date/Time	Received by (Signature and Company)	Date/Time	Sample Condition upon Receipt
Relinquished by (Signature and Company)	Date/Time	Received by (Signature and Company)	Date/Time	Sample Condition upon Receipt
April 12, 2010 Copies: Pink -	Retained by San	nple Coordinator; Yellow - Retained by laboratory; Whit	te - Included in ana	lytical report Page of

### APPENDIX F Libby Asbestos Project Record of Modification Forms (Field and Laboratory)



### Record of Modification to Documents Governing Field Activities Libby Asbestos Project

Form No. LFO-000\_\_\_

Instructions to Requester: Email draft modification form to the contacts at bottom of form for review and approval. File approved copy with the CDM Quality Assurance Coordinator (QAC) at the Libby Field Office (LFO). The QAC will distribute approved copies and maintain the originals at the LFO.

Requester:			Title:					
	pany: Date:							
Governing document (title and approved date) or SOP (title and SOP number):								
Field logbook and pa	age number where mo	odification is docume	ented (or attach associ	ated correspondence):				
			ssary; include revised	text for all document or				
Implication(s) of mod	dification (if applicable	e, attach a list of affe	cted property addresse	es or sample IDs):				
Duration of modificat	tion (indicate one):							
Temporary	Date(s):		-					
Permanent	Effective Date:		_					
Data Quality Indicato indicators):	or (indicate one; refer	ence the definitions	below for direction on s	selecting data quality				
□ Not Applic	able	☐ Low Bias		☐ High Bias				
☐ Reject		☐ Estimate		☐ No Bias				
CDM Technical Revi (CDM Project Manag	ew and Approval: ger or designate)			Date:				
EPA Review and Ap (USEPA RPM or des	proval:signate)		Date:_					

#### DATA QUALITY INDICATOR DEFINITIONS

**Reject** - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

**Low Bias** - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

**Estimate** - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

**High Bias** - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

**No Bias** - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.



### **Request for Modification**

### Laboratory Activities

LB-\_\_\_\_

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.

File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

	se applicable): TEN 00/R-93/116 AST	ΓM D5755	EPA/540/2-9		0 NIOSH 9002 SRC-LIBBY-03
Requester:			Title	e:	
Company:			Dat	e:	
Description of Modifica	tion:				
Reason for Modification	n:				
Potential Implications of	of this Modification:				
Laboratory Applicability	/ (circle one): All	Individual(s	3)		
This laboratory modific	ation is (circle one):	NEW APPE	NDS to	SUP	PERCEDES
	n (circle one): Date(s): Analytical Batch ID: ation Forms – Attach leg	. <u> </u>			nckages
Permanent Permanent Modific	(Complete Propose cation Forms – Maintain I				
Data Quality Indicator	(circle one) – Please	reference definiti	ons on reverse sid	e for direction on s	electing data quality indicator
Not Applicable	Reject	Low Bias	Estimate	High Bias	No Bias
Proposed Modification when applicable):	to Method (attach a	dditional sheets	s if necessary; s	tate section and	page numbers of Method
Technical Review:					Date:
(L	aboratory Manager or				
Project Review and Ap	proval:(Volne: Pro	niect Technical I	ead or designate)		Date:
Approved By:	(Voipo. 176	,,000 10011111001 E	oud of doorgridto)		Date:

(USEPA: Project Chemist or designate)

#### DATA QUALITY INDICATOR DEFINITIONS

**Reject** - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

**Low Bias** - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

**Estimate** - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

*High Bias* - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

**No Bias** - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.